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PASSWORD:

TERMINAL (ENTER 1, 2, 3, OR ?):2

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NEWS NEWS	1 2	AUG	10	Web Page for STN Seminar Schedule - N. America Time limit for inactive STN sessions doubles to 40
				minutes
NEWS	3	AUG	18	COMPENDEX indexing changed for the Corporate Source (CS) field
NEWS	4	AUG	24	ENCOMPLIT/ENCOMPLIT2 reloaded and enhanced
NEWS	5	AUG		CA/CAplus enhanced with legal status information for U.S. patents
NEWS	6	SEP	09	50 Millionth Unique Chemical Substance Recorded in CAS REGISTRY
NEWS	7	SEP	11	WPIDS, WPINDEX, and WPIX now include Japanese FTERM thesaurus
NEWS	8	OCT	21	Derwent World Patents Index Coverage of Indian and Taiwanese Content Expanded
NEWS	9	OCT	21	Derwent World Patents Index enhanced with human translated claims for Chinese Applications and Utility Models
NEWS	10	NOV	23	Addition of SCAN format to selected STN databases
NEWS	11	NOV	23	Annual Reload of IFI Databases
NEWS	12	DEC	01	FRFULL Content and Search Enhancements
NEWS	13	DEC	01	DGENE, USGENE, and PCTGEN: new percent identity feature for sorting BLAST answer sets
NEWS	14	DEC	02	Derwent World Patent Index: Japanese FI-TERM thesaurus added
NEWS	15	DEC	02	PCTGEN enhanced with patent family and legal status display data from INPADOCDB
NEWS	16	DEC	02	USGENE: Enhanced coverage of bibliographic and sequence information
NEWS	17	DEC	21	New Indicator Identifies Multiple Basic Patent Records Containing Equivalent Chemical Indexing in CA/CAplus
NEWS	18	JAN	12	Match STN Content and Features to Your Information Needs, Quickly and Conveniently
NEWS	19	JAN	25	Annual Reload of MEDLINE database
NEWS	20	FEB	16	STN Express Maintenance Release, Version 8.4.2, Is Now Available for Download
NEWS	21	FEB	16	Derwent World Patents Index (DWPI) Revises Indexing of Author Abstracts
NEWS	22	FEB	16	New FASTA Display Formats Added to USGENE and PCTGEN
NEWS	23	FEB	16	INPADOCDB and INPAFAMDB Enriched with New Content and Features
NEWS	24	FEB	16	INSPEC Adding Its Own IPC codes and Author's E-mail Addresses

NEWS EXPRESS FEBRUARY 15 10 CURRENT WINDOWS VERSION IS V8.4.2, AND CURRENT DISCOVER FILE IS DATED 15 JANUARY 2010.

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FILE 'HOME' ENTERED AT 17:13:59 ON 18 MAR 2010

=> fil reg
COST IN U.S. DOLLARS

SINCE FILE TOTAL ENTRY SESSION 0.44 0.44

FULL ESTIMATED COST

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STRUCTURE FILE UPDATES: 17 MAR 2010 HIGHEST RN 1211109-76-0 DICTIONARY FILE UPDATES: 17 MAR 2010 HIGHEST RN 1211109-76-0

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TSCA INFORMATION NOW CURRENT THROUGH January 8, 2010.

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http://www.cas.org/support/stngen/stndoc/properties.html

=>

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r'hr'h

chain nodes :

14

ring nodes :

1 2 3 4 5 6 7 8 9 10 11 12 13

chain bonds :

12 - 14

ring bonds :

 $1-2 \quad 1-6 \quad 2-3 \quad 3-4 \quad 4-5 \quad 5-6 \quad 5-7 \quad 6-10 \quad 7-8 \quad 8-9 \quad 8-11 \quad 9-10 \quad 9-13 \quad 11-12 \quad 12-13$ 

exact/norm bonds :

1-2 1-6 2-3 3-4 4-5 5-6 5-7 6-10 7-8 8-9 9-10 11-12 12-13 12-14

exact bonds : 8-11 9-13

isolated ring systems :

containing 1 :

G1:H,Ak

Match level :

1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:Atom 7:Atom 8:Atom 9:Atom 10:Atom

11:Atom 12:Atom 13:Atom 14:CLASS

L1 STRUCTURE UPLOADED

=> s l1 sss full

FULL SEARCH INITIATED 17:15:20 FILE 'REGISTRY'

FULL SCREEN SEARCH COMPLETED - 22872 TO ITERATE

100.0% PROCESSED 22872 ITERATIONS

84 ANSWERS

SEARCH TIME: 00.00.01

L2 84 SEA SSS FUL L1

=> fil cap

COST IN U.S. DOLLARS SINCE FILE TOTAL

FULL ESTIMATED COST ENTRY SESSION 191.54 191.98

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FILE COVERS 1907 - 18 Mar 2010 VOL 152 ISS 12 FILE LAST UPDATED: 17 Mar 2010 (20100317/ED) REVISED CLASS FIELDS (/NCL) LAST RELOADED: Dec 2009 USPTO MANUAL OF CLASSIFICATIONS THESAURUS ISSUE DATE: Dec 2009

CAplus now includes complete International Patent Classification (IPC) reclassification data for the first quarter of 2010.

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This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s 11

REG1stRY INITIATED

Substance data SEARCH and crossover from CAS REGISTRY in progress... Use DISPLAY HITSTR (or FHITSTR) to directly view retrieved structures.

6 ANSWERS

SAMPLE SEARCH INITIATED 17:15:36 FILE 'REGISTRY'
SAMPLE SCREEN SEARCH COMPLETED - 1158 TO ITERATE

100.0% PROCESSED 1158 ITERATIONS

SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE \*\*COMPLETE\*\*

BATCH \*\*COMPLETE\*\*

PROJECTED ITERATIONS: 21119 TO 25201

PROJECTED ANSWERS: 6 TO 266

L3 6 SEA SSS SAM L1

L4 8 L3

=> fil cap COST IN U.S. DOLLARS

COST IN U.S. DOLLARS
SINCE FILE TOTAL
ENTRY SESSION
FULL ESTIMATED COST
0.50
193.47

FILE 'CAPLUS' ENTERED AT 17:15:39 ON 18 MAR 2010 USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT. PLEASE SEE "HELP USAGETERMS" FOR DETAILS.

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FILE COVERS 1907 - 18 Mar 2010 VOL 152 ISS 12
FILE LAST UPDATED: 17 Mar 2010 (20100317/ED)
REVISED CLASS FIELDS (/NCL) LAST RELOADED: Dec 2009
USPTO MANUAL OF CLASSIFICATIONS THESAURUS ISSUE DATE: Dec 2009

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=> dhis

DHIS IS NOT A RECOGNIZED COMMAND

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=> d his

(FILE 'HOME' ENTERED AT 17:13:59 ON 18 MAR 2010)

FILE 'REGISTRY' ENTERED AT 17:14:59 ON 18 MAR 2010

L1 STRUCTURE UPLOADED

L2 84 S L1 SSS FULL

FILE 'CAPLUS' ENTERED AT 17:15:33 ON 18 MAR 2010 S L1

FILE 'REGISTRY' ENTERED AT 17:15:35 ON 18 MAR 2010 L3 6 S L1

FILE 'CAPLUS' ENTERED AT 17:15:36 ON 18 MAR 2010 L4 8 S L3

FILE 'CAPLUS' ENTERED AT 17:15:39 ON 18 MAR 2010

=> s 11 and (pry<2005 or py<2005)
 REG1stRY INITIATED</pre>

Substance data SEARCH and crossover from CAS REGISTRY in progress... Use DISPLAY HITSTR (or FHITSTR) to directly view retrieved structures.

SAMPLE SEARCH INITIATED 17:16:21 FILE 'REGISTRY'
SAMPLE SCREEN SEARCH COMPLETED - 1158 TO ITERATE

100.0% PROCESSED 1158 ITERATIONS 6 ANSWERS

SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE \*\*COMPLETE\*\*
BATCH \*\*COMPLETE\*\*
PROJECTED ITERATIONS: 21119 TO 25201
PROJECTED ANSWERS: 6 TO 266

L5 6 SEA SSS SAM L1

L6 8 L5

4640932 PRY<2005 25157538 PY<2005

L7 8 L6 AND (PRY<2005 OR PY<2005)

=> fil cap

COST IN U.S. DOLLARS SINCE FILE TOTAL

ENTRY SESSION 5.12 199.58

FULL ESTIMATED COST

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FILE COVERS 1907 - 18 Mar 2010 VOL 152 ISS 12 FILE LAST UPDATED: 17 Mar 2010 (20100317/ED) REVISED CLASS FIELDS (/NCL) LAST RELOADED: Dec 2009 USPTO MANUAL OF CLASSIFICATIONS THESAURUS ISSUE DATE: Dec 2009

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=> d his

(FILE 'HOME' ENTERED AT 17:13:59 ON 18 MAR 2010)

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T.1
                 STRUCTURE UPLOADED
T.2
              84 S L1 SSS FULL
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     FILE 'REGISTRY' ENTERED AT 17:15:35 ON 18 MAR 2010
L3
               6 S L1
     FILE 'CAPLUS' ENTERED AT 17:15:36 ON 18 MAR 2010
L4
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L5
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L6
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L7
               8 S L6 AND (PRY<2005 OR PY<2005)
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=> s 12
            32 L2
L8
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            32 L2
       4640932 PRY<2005
      25157538 PY<2005
L9
            28 L2 AND (PRY<2005 OR PY<2005)
=> d 1-28 ibib abs hitstr
    ANSWER 1 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN
ACCESSION NUMBER:
                     2004:1080987 CAPLUS
DOCUMENT NUMBER:
                          142:58224
TITLE:
                          Heterocyclic colorants based on diazabenzoisoindoles.
                          Heckmann, Heino; Metz, Hans Joachim
INVENTOR(S):
PATENT ASSIGNEE(S):
                         Clariant G.m.b.H., Germany
SOURCE:
                          PCT Int. Appl., 31 pp.
                          CODEN: PIXXD2
DOCUMENT TYPE:
                          Patent
LANGUAGE:
                          German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:
                   KIND DATE APPLICATION NO. DATE
     PATENT NO.
     _____
                          ____
                                  _____
                                               ______
                          A1 20041216 WO 2004-EP5459 20040521 <--
     WO 2004108836
         W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH,
             CN, CO, CR, CU, CZ, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO,
             NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ,
              TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW
         RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM,
             AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE,
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SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE,

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EP	1639	047			В1		2008	0423										
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		ΙE,	SI,	FΙ,	RO,	CY,	TR,	BG,	CZ,	EE	, HU,	PL,	SK					
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JP	2006	5272	86		T		2006	1130		JΡ	2006-	5157	83		2	0040	521	<
US	2007	0264	600		A1		2007	1115	1	US	2007-	5600	95		2	0070	323	<
PRIORIT	Y APP	LN.	INFO	.:						DE	2003-	1032	6211	Ž	A 2	0030	611	<
									1	WO	2004-	EP54	59	I	W 2	0040	521	<
OTHER SO	OURCE	(S):			MAR:	PAT	142:	5822	4									

- AB Diazabenzoisoindoles I (A = aliphatic or heterocyclic carbonyl-containing fragments, B = optionally substituted ortho-C6-18 aryls) are useful in paints and printing inks with an alkyd resins binder. Thus, a pigment II [prepared by treating 1-amino-1-(2,4,6-trioxotetrahydropyrimidine-5-yl)-3-(2,4,6-trioxotetrahydropyrimidine-5-ylidene)-4,9-diazabenzo[f]isoindole with H2SO4] is used with melamine-based alkyd resins binder as an yellow paint.
- IT 808134-07-8P 808134-08-9P RL: IMF (Industrial manufacture); RCT (Reactant); PREP (Preparation); RACT (Reactant or reagent)
  - (diazabenzoisoindole pigments for paints and printing inks)
- RN 808134-07-8 CAPLUS
- CN 2,4,6(1H,3H,5H)-Pyrimidinetrione, 5-[1-amino-2,3-dihydro-3-(tetrahydro-2,4,6-trioxo-5(2H)-pyrimidinylidene)-1H-pyrrolo[3,4-b]quinoxalin-1-yl]-(9CI) (CA INDEX NAME)

RN 808134-08-9 CAPLUS

CN 2,4,6(1H,3H,5H)-Pyrimidinetrione, 5-[1-amino-2,3-dihydro-3-(tetrahydro-1,3-dimethyl-2,4,6-trioxo-5(2H)-pyrimidinylidene)-1H-pyrrolo[3,4-b]quinoxalin-1-yl]-1,3-dimethyl- (9CI) (CA INDEX NAME)

IT 808134-09-0P 808134-10-3P 808134-11-4P 808134-12-5P 808134-13-6P 808134-14-7P

RL: IMF (Industrial manufacture); TEM (Technical or engineered material use); PREP (Preparation); USES (Uses)

(diazabenzoisoindole pigments for paints and printing inks)

RN 808134-09-0 CAPLUS

CN 1H-Indene-1,3(2H)-dione, 2-[3-amino-3-(2,3-dihydro-1,3-dioxo-1H-inden-2-yl)-2,3-dihydro-1H-pyrrolo[3,4-b]quinoxalin-1-ylidene]- (9CI) (CA INDEX NAME)

RN 808134-10-3 CAPLUS

CN Pyrimido[1,2-a]benzimidazole-2,4(1H,3H)-dione,
3-[1-amino-3-(1,2-dihydro-2,4-dioxopyrimido[1,2-a]benzimidazol-3(4H)ylidene)-2,3-dihydro-1H-pyrrolo[3,4-b]quinoxalin-1-yl]- (9CI) (CA INDEX NAME)

RN 808134-11-4 CAPLUS

CN 2,4,6(1H,3H,5H)-Pyrimidinetrione, 5,5'-(1H-pyrrolo[3,4-b]quinoxaline-1,3(2H)-diylidene)bis- (9CI) (CA INDEX NAME)

RN 808134-12-5 CAPLUS

CN 2,4,6(1H,3H,5H)-Pyrimidinetrione, 5,5'-(1H-pyrrolo[3,4-b]quinoxaline-1,3(2H)-diylidene)bis[1,3-dimethyl- (9CI) (CA INDEX NAME)

RN 808134-13-6 CAPLUS

CN 1H-Indene-1,3(2H)-dione, 2,2'-(1H-pyrrolo[3,4-b]quinoxaline-1,3(2H)-diylidene)bis- (9CI) (CA INDEX NAME)

RN 808134-14-7 CAPLUS

CN Pyrimido[1,2-a]benzimidazole-2,4-dione, 3,3'-(1H-pyrrolo[3,4-b]quinoxaline-1,3(2H)-diylidene)bis- (9CI) (CA INDEX NAME)

REFERENCE COUNT:

4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 2 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN ACCESSION NUMBER: 2002:831366 CAPLUS

DOCUMENT NUMBER: 138:255021

TITLE: Synthesis and nucleophilic opening of a new C2

symmetric bis-aziridine. First synthesis of aziridines

using polymer-supported triphenylphosphine

AUTHOR(S): McCort, Isabelle; Ballereau, Stephanie; Dureault,

Annie; Depezay, Jean-Claude

CORPORATE SOURCE: Laboratoire de Chimie et Biochimie Pharmacologiques et

Toxicologiques associe au CNRS, Universite Rene

Descartes, Paris, 75270, Fr.

SOURCE: Tetrahedron (2002), 58(44), 8947-8955

CODEN: TETRAB; ISSN: 0040-4020

PUBLISHER: Elsevier Science Ltd.

DOCUMENT TYPE: Journal LANGUAGE: English

OTHER SOURCE(S): CASREACT 138:255021

The synthesis of (2S,2'S)-2,3-bis(2-aziridinyl)quinoxaline from 1,2:5,6-bis-O-(1-methylethylidene)-D-threo-3,4-hexodiulose (a D-mannitol derivative) is reported. Reductive aminocyclization of diazido diols has been achieved by polymer-supported PPh3 in a suitable manner. An N-Boc derivative [i.e., (2S,2'S)-2,3-bis(1-BOC-2-aziridinyl)quinoxaline] and N-tosyl derivative [i./e., (2S,2'S)-2,3-bis(1-tosyl-2-aziridinyl)quinoxaline] were treated with different nucleophiles either in protic or aprotic media.

IT 502699-52-7P 502699-54-9P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation and nucleophilic opening of C2 sym. bis-aziridine; first synthesis of aziridines using polymer-supported triphenylphosphine)

RN 502699-52-7 CAPLUS

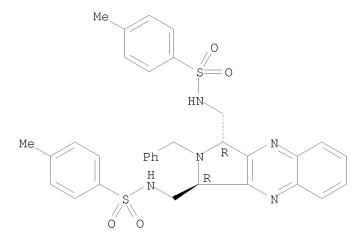
CN Carbamic acid, [[(1R,3R)-2,3-dihydro-2-(phenylmethyl)-1H-pyrrolo[3,4-b]quinoxaline-1,3-diyl]bis(methylene)]bis-, bis(1,1-dimethylethyl) ester (9CI) (CA INDEX NAME)

Absolute stereochemistry.

RN 502699-54-9 CAPLUS

CN Benzenesulfonamide, N,N'-[[(1R,3R)-2,3-dihydro-2-(phenylmethyl)-1H-pyrrolo[3,4-b]quinoxaline-1,3-diyl]bis(methylene)]bis[4-methyl- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



OS.CITING REF COUNT: 5 THERE ARE 5 CAPLUS RECORDS THAT CITE THIS RECORD

(5 CITINGS)

REFERENCE COUNT: 19 THERE ARE 19 CITED REFERENCES AVAILABLE FOR THIS

RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 3 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1999:737917 CAPLUS

DOCUMENT NUMBER: 132:93285

TITLE: Synthesis of new fluorine-containing derivatives of

quinoxaline 1,4-dioxides and condensed systems derived

from them

AUTHOR(S): Chupakhin, O. N.; Kotovskaya, S. K.; Perova, N. M.;

Baskakova, Z. M.; Charushin, V. N.

CORPORATE SOURCE: Ural's State Technical University, Yekaterinburg,

620002, Russia

SOURCE: Chemistry of Heterocyclic Compounds (New

York) (Translation of Khimiya Geterotsiklicheskikh

Soedinenii) (1999), 35(4), 459-469 CODEN: CHCCAL; ISSN: 0009-3122

PUBLISHER: Consultants Bureau

DOCUMENT TYPE: Journal LANGUAGE: English

OTHER SOURCE(S): CASREACT 132:93285

AB The Beirut reaction of 5,6-difluorobenzofuroxan with 1,3-diketones,

 $\beta\text{-keto}$  esters, and  $\beta\text{-keto}$  amides produces

6,7-difluoroquinoxaline 1,4-dioxides. The condensation of

2-ethoxycarbonyl-6,7-difluoro-3-methylquinoxaline 1,4-dioxide is studied. Fluorinated furo[3,4-b]- and pyrrolo[3,4-b]quinoxaline 4,9-dioxides are synthesized and further functionalized by nucleophilic substitution of fluorine and reduction of the N-O bond.

IT 230948-44-4P 254755-08-3P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(fluorinated quinoxaline 1,4-dioxides and condensed systems derived from them)

RN 230948-44-4 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one,

7-fluoro-2,3-dihydro-2-methyl-6-(methylamino)-, 4,9-dioxide (CA INDEX NAME)

RN 254755-08-3 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 2-ethyl-6,7-difluoro-2,3-dihydro-, 4,9-dioxide (CA INDEX NAME)

IT 230948-41-1P 230948-45-5P 254755-09-4P

254755-10-7P 254755-11-8P 254755-13-0P
RL: SPN (Synthetic preparation); PREP (Preparation)
 (fluorinated quinoxaline 1,4-dioxides and condensed systems derived from them)

RN 230948-41-1 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 6,7-difluoro-2,3-dihydro-, 4,9-dioxide (CA INDEX NAME)

RN 230948-45-5 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 2-ethyl-6-(ethylamino)-7-fluoro-2,3-dihydro-, 4,9-dioxide (CA INDEX NAME)

RN 254755-09-4 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 6,7-difluoro-2,3-dihydro-2-(2-hydroxyethyl)-, 4,9-dioxide (CA INDEX NAME)

RN 254755-10-7 CAPLUS

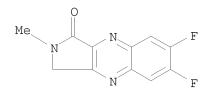
CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 2-ethyl-7-fluoro-2,3-dihydro-6-(4-morpholinyl)-, 4,9-dioxide (CA INDEX NAME)

RN 254755-11-8 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 2-ethyl-6,7-difluoro-2,3-dihydro- (CA INDEX NAME)

RN 254755-13-0 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 6,7-difluoro-2,3-dihydro-2-methyl- (CA INDEX NAME)



OS.CITING REF COUNT: 1 THERE ARE 1 CAPLUS RECORDS THAT CITE THIS RECORD

(1 CITINGS)

REFERENCE COUNT: 15 THERE ARE 15 CITED REFERENCES AVAILABLE FOR THIS

RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 4 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1999:238166 CAPLUS

DOCUMENT NUMBER: 131:102256

TITLE: Synthesis of fluorinated furo- and

pyrrolo[3,4-b]quinoxaline 4,9-dioxides

AUTHOR(S): Kotovskaya, Svetlana K.; Perova, Natalya M.;

Charushin, Valery N.; Chupakhin, Oleg N.

CORPORATE SOURCE: Department of Chemistry, Urals State Technical

University, Yekaterinburg, 620002, Russia

SOURCE: Mendeleev Communications (1999), (2), 76-77

CODEN: MENCEX; ISSN: 0959-9436 Russian Academy of Sciences

PUBLISHER: Russian Acade
DOCUMENT TYPE: Journal

LANGUAGE: Sournai English

GΙ

$$\begin{array}{c|c} & & & & \\ & & & & \\ & & \\ & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & &$$

The reaction of 5,6-difluorobenzofuroxane I with Et acetoacetate in the presence of triethylamine results in the formation of 2-methyl-3-ethoxycarbonyl-6,7-difluoroquinoxaline 1,4-dioxide which was converted consequently into the bromomethyl II (R = Br) and acetoxymethyl II (R = OAc) derivs.; hydrolysis of the latter with hydrochloric acid gave furo[3,4-b]quinoxaline 4,9-dioxide III (X = O, Y = F). Compound II (R = Br) was transformed by the action of ammonia and primary alkyl amines into 2-substituted 1,3-dihydro-2H-pyrrolo[3,4-b]quinoxaline 4,9-dioxides III (X = NR', R' = H, cyclohexyl) and further into the corresponding 6-amino compds. III (X = cyclohexylamino, Y = morpholino; X = NR', Y = NHR', R' =

Me, Et).

IT 230948-41-1P 230948-44-4P 230948-45-5P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of fluorinated furo- and pyrroloquinoxaline dioxides)

RN 230948-41-1 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 6,7-difluoro-2,3-dihydro-, 4,9-dioxide (CA INDEX NAME)

RN 230948-44-4 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 7-fluoro-2,3-dihydro-2-methyl-6-(methylamino)-, 4,9-dioxide (CA INDEX NAME)

RN 230948-45-5 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 2-ethyl-6-(ethylamino)-7-fluoro-2,3-dihydro-,4,9-dioxide (CA INDEX NAME)

OS.CITING REF COUNT: 7 THERE ARE 7 CAPLUS RECORDS THAT CITE THIS RECORD

(7 CITINGS)

REFERENCE COUNT: 12 THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 5 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1994:101292 CAPLUS

DOCUMENT NUMBER: 120:101292

ORIGINAL REFERENCE NO.: 120:17823a,17826a

TITLE: Water-soluble tetraazaporphines and fluorochromes for

labeling

INVENTOR(S): Tai, Seiji; Katayose, Mitsuo; Watanabe, Hiroo

PATENT ASSIGNEE(S): Hitachi Chemical Co., Ltd., Japan

SOURCE: Eur. Pat. Appl., 110 pp.

CODEN: EPXXDW

DOCUMENT TYPE: Patent LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.		DATE
				-	
EP 502723	A2	19920909	EP 1992-301873		19920304 <
EP 502723	A3	19930127			
EP 502723	В1	19961009			
R: DE, FR, GB,	IT, NL				
JP 05163439	A	19930629	JP 1992-22192		19920207 <
JP 2964761	В2	19991018			
US 5438135	A	19950801	US 1992-846169		19920305 <
PRIORITY APPLN. INFO.:			JP 1991-38349	Α	19910305 <
			JP 1991-146005	Α	19910618 <
			JP 1991-159308	Α	19910701 <
			JP 1991-268016	Α	19911017 <

ASSIGNMENT HISTORY FOR US PATENT AVAILABLE IN LSUS DISPLAY FORMAT OTHER SOURCE(S): MARPAT 120:101292

AB Water-soluble tetraazaporphines, fluorochromes from them, biol. substances labeled with the fluorochromes, reagents comprising them, and their use in fluorescence anal. are described. A semiconductor laser having an output wavelength of 670-840 nm is used as a light source. Na bis(tributylsilyloxy)silicon

tetraphenylthio(naphthalocyanine)octacarboxylate (I) (preparation described) was coupled to the 5'-end of ACACAACTGTGTTCACTAGC and used in the detection of the  $\beta$ -globin gene in human DNA. I was also coupled to PABA and morphine. Antimorphine monoclonal antibody had only slightly diminished affinity for the morphine conjugate.

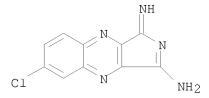
IT 145614-76-2P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation and reaction of, in tetraazaporphine fluorochrome label preparation)

RN 145614-76-2 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-3-amine, 6-chloro-1-imino- (CA INDEX NAME)



OS.CITING REF COUNT: 4 THERE ARE 4 CAPLUS RECORDS THAT CITE THIS RECORD (4 CITINGS)

L9 ANSWER 6 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1981:30470 CAPLUS

DOCUMENT NUMBER: 94:30470
ORIGINAL REFERENCE NO.: 94:5019a,5022a

TITLE: Synthesis of quinoxaline- and indole-2,3-dicarboxylic

acid imides

Augustin, M.; Koehler, M.; Faust, J.; Al-Holly, M. M. AUTHOR(S): CORPORATE SOURCE:

Sekt. Chem., Martin Luther Univ., Halle-Wittenberg,

DDR-402, Ger. Dem. Rep.

SOURCE: Tetrahedron (1980), 36(12), 1801-5

CODEN: TETRAB; ISSN: 0040-4020

DOCUMENT TYPE: Journal LANGUAGE: German

OTHER SOURCE(S): CASREACT 94:30470

GΙ

The maleimides I (R = H, R1 = C1, R2R3 = NPh, NMe) reacted with NaN3 AB (Me2CO/H2O, room temperature, 10 min) to give the indole-2,3-dicarboxylic acid imides I (RR1 = NH, R2R3 = NPh, NMe) (33 and 34%, resp.). These reacted readily with nucleophiles to give a range of 6-methylindole-2,3-dicarboxylic acid derivs. in high yield (53-92%). E.g., I (RR1 = NH, R2R3 = NPh) with MeOH (10 min) gave 90% I (RR1 = NH, R2 = NHPh, R3 = OMe). The quinoxaline-2,3-dicarboxylic acid imides II (R =Ph, Me) were prepared similarly. Treatment of II (R = Ph) with aqueous NH3

the intermediate III (R = CO2NH2) which either hydrolyzed to the acid-amide or decarboxylated to the crystalline monoamide III (R = H).

76039-54-8P ΙT

gave

RL: SPN (Synthetic preparation); PREP (Preparation) (preparation of, by cyclization of maleimide)

RN 76039-54-8 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxaline-1,3(2H)-dione, 2-methyl- (CA INDEX NAME)

OS.CITING REF COUNT: 4 THERE ARE 4 CAPLUS RECORDS THAT CITE THIS RECORD (5 CITINGS)

ANSWER 7 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN L9

ACCESSION NUMBER: 1979:611366 CAPLUS

DOCUMENT NUMBER: 91:211366

ORIGINAL REFERENCE NO.: 91:34061a,34064a

TITLE: Structure and reactivity of isoannelated heterocyclic

systems with  $4n\pi$ - and  $(4n+2)\pi$ -electrons. 7.

2-tert-Butylpyrrolo[3,4-b]quinoxaline. Synthesis,

properties, reactions

AUTHOR(S): Kreher, Richard; Use, Goetz

CORPORATE SOURCE: Inst. Org. Chem., Tech. Hochsch. Darmstadt, Darmstadt,

Fed. Rep. Ger.

SOURCE: Tetrahedron Letters (1978), (47), 4671-4

CODEN: TELEAY; ISSN: 0040-4039

DOCUMENT TYPE: Journal LANGUAGE: German

OTHER SOURCE(S): CASREACT 91:211366

GΙ

The title pyrroloquinoxaline (I; R = R2 = H, R1 = CMe3) (II) was prepared from the corresponding 1,3-dihydro compound by MnO2 oxidation in C6H6 or by sequential treatment with NaOH-MeOH and O. II underwent cycloaddn. reactions with N-methylmaleimide and di-Me acetylenedicarboxylate, addition reactions with di-Me azodicarboxylate [to give I (R = MeO2CNHNCO2Me, R2 = H, MeO2CNHNCO2Me, R1 = CMe3)], and alkylation at N-4.

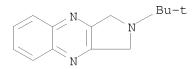
IT 70200-39-4P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of, as intermediate in tert-butylpyrroloquinoxaline preparation)

RN 70200-39-4 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxaline, 2-(1,1-dimethylethyl)-2,3-dihydro- (CA INDEX NAME)



OS.CITING REF COUNT: 4 THERE ARE 4 CAPLUS RECORDS THAT CITE THIS RECORD (4 CITINGS)

L9 ANSWER 8 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1979:87445 CAPLUS

DOCUMENT NUMBER: 90:87445

ORIGINAL REFERENCE NO.: 90:13865a,13868a

TITLE: Derivatives of quinoxalino-[2,3-c]pyrroline

INVENTOR(S): Hahn, Witold; Lesiak, Jerzy PATENT ASSIGNEE(S): Uniwersytet Lodzki, Pol.

SOURCE: Pol., 2 pp.

CODEN: POXXA7

DOCUMENT TYPE: Patent LANGUAGE: Polish

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE

PL 71049 PRIORITY APPLN. INFO.: Α5 19740430 PL 1972-155806 PL 1972-155806

19720605 <--A 19720605 <--

GT

AΒ The title compds. (I; R = R1 = Me, Et, Pr, Bu, allyl, CH2CH2OH; R = Me, R1 = CH2Ph; NRR1 = pyrrolidino, morpholino) were prepared by heating EtOH solns. of 2,3-bis(bromomethyl)quinoxaline with 2 equiv RR1NH for .apprx.8 h at  $20-100^{\circ}$  (usually  $30-50^{\circ}$ ).

40197-24-8P ΙT

40197-25-9P 40197-26-0P

40197-27-1P

40197-28-2P 40197-29-3P

40197-30-6P

RL: SPN (Synthetic preparation); PREP (Preparation)

Ι

(preparation of)

RN 40197-24-8 CAPLUS

1H-Pyrrolo[3,4-b]quinoxalinium, 2,3-dihydro-2,2-dimethyl-, bromide (1:1) CN (CA INDEX NAME)

● <sub>Br</sub>-

RN 40197-25-9 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalinium, 2,2-diethyl-2,3-dihydro-, bromide (1:1) (CA INDEX NAME)

● Br-

RN 40197-26-0 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalinium, 2,3-dihydro-2,2-dipropyl-, bromide (1:1) (CA INDEX NAME)

• Br-

RN 40197-27-1 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalinium, 2,2-dibutyl-2,3-dihydro-, bromide (1:1) (CA INDEX NAME)

● Br-

RN 40197-28-2 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalinium, 2,3-dihydro-2,2-bis(2-hydroxyethyl)-, bromide (1:1) (CA INDEX NAME)

• Br-

RN 40197-29-3 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalinium, 2,3-dihydro-2,2-di-2-propen-1-yl-, bromide (1:1) (CA INDEX NAME)

$$CH_2-CH=CH_2$$
 $N$ 
 $+$ 
 $CH_2-CH=CH_2$ 

• Br-

RN 40197-30-6 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalinium, 2,3-dihydro-2-methyl-2-(phenylmethyl)-, bromide (1:1) (CA INDEX NAME)

$$\begin{array}{c|c} & \text{Me} \\ \hline & \\ N \\ & + \\ \text{CH}_2 - \text{Ph} \end{array}$$

• Br-

L9 ANSWER 9 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1979:81450 CAPLUS

DOCUMENT NUMBER: 90:81450

ORIGINAL REFERENCE NO.: 90:12825a,12828a

TITLE: Some relations between the structure, and

antibacterial and growth-stimulating effects of

heterocyclic compounds

AUTHOR(S): Novacek, L.; Belusa, J.; Hruskova, V.; Vavrinova, D. CORPORATE SOURCE: Vyzk. Ustav Cistych Chem. N. P. Lachema, Brno, Czech.

SOURCE: Cesko-Slovenska Farmacie (1978), 27(4),

173-7

CODEN: CKFRAY; ISSN: 0009-0530

DOCUMENT TYPE: Journal LANGUAGE: Czech

OTHER SOURCE(S): CASREACT 90:81450

GΙ

Derivs. of 2,3-dihydro-1-oxo-1H-pyrrolo[3,4-b]quinoxaline 4,9-dioxide, quinazoline N-oxide, 3,4-dihydro-4-oxoquinazoline, and 4-thiazolidinecarboxylic acid were synthesized and examined for their antibacterial activities and their ability to promote the growth of farm animals. 2-(5-Nitrofuryl)quinazoline 1,3-dioxide (I) [65884-43-7] and

1-hydroxy-2-(5-nitrofuryl)-1,2-dihydroquinazoline 3-oxide [69020-13-9] were the most active inhibitors of Escherichia coli, and 1-hydroxy-2-methyl-1,2-dihydroquinazoline 3-oxide [25509-11-9] was the most effective in improved weight gain and feedstuff conversion. Growth promoters should have some antibacterial activity, but excessive activity is not desirable.

IT 40970-22-7P 65993-95-5P

RL: BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); SPN (Synthetic preparation); THU (Therapeutic use); BIOL (Biological study); PREP (Preparation); USES (Uses)

(preparation and bactericidal activity of, animal growth promotion in relation to)

RN 40970-22-7 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 2,3-dihydro-2-methyl-, 4,9-dioxide (CA INDEX NAME)

RN 65993-95-5 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 2,3-dihydro-2-(2-hydroxyethyl)-, 4,9-dioxide (CA INDEX NAME)

L9 ANSWER 10 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1978:151119 CAPLUS

DOCUMENT NUMBER: 88:151119

ORIGINAL REFERENCE NO.: 88:23803a,23806a

TITLE: Study of new growth stimulators. Part 1. Basic testing of the original substances in chickens

AUTHOR(S): Broz, Jiri; Sevcik, Bohumil

CORPORATE SOURCE: Vyzk. Ustav Biofakt. Vet. Leciva, Pohori-Chotoun,

Czech.

SOURCE: Biologizace a Chemizace Vyzivy Zvirat (1977

), 13(4), 357-74

CODEN: BCVZB4; ISSN: 0523-6738

DOCUMENT TYPE: Journal LANGUAGE: Czech

GΙ

In screening studies with chicks at 50-100 ppm of test substances added to AΒ a practical-type ration, growth stimulation was observed with 19 quinoxaline-1,4-dioxides, especially VUFB 11803 (I) [65870-53-3], VUFB 11502 [65884-46-0], VUFB 11486 [65993-94-4], VUFB 11495 [65884-47-1], VUFB 11806 [65884-48-2], VUFB 11815 [65884-49-3], and VUFB 9960 [65884-50-6], with 5 quinoxaline-1,3-dioxides, with nitrofurans, with ethanolamides of fatty acids, and with several other com. substances, e.g., 3-oxauracil [5638-70-0] and cyproheptadine [129-03-3]. ΙT 65993-95-5

RL: BIOL (Biological study)

(growth stimulant, for chicken)

RN 65993-95-5 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 2,3-dihydro-2-(2-hydroxyethyl)-, 4,9-dioxide (CA INDEX NAME)

ANSWER 11 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1976:577483 CAPLUS

DOCUMENT NUMBER: 85:177483

ORIGINAL REFERENCE NO.: 85:28371a,28374a

TITLE: Quinoxalino[2,3-c]pyrroles INVENTOR(S): Hahn, Witold; Lesiak, Jerzy PATENT ASSIGNEE(S): Uniwersytet Lodzki, Pol.

Pol., 2 pp. SOURCE: CODEN: POXXA7

DOCUMENT TYPE: Patent LANGUAGE: Polish

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

GΙ

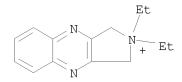
PATENT NO. KIND DATE APPLICATION NO. DATE PL 71049 19751201 PL 1973-155806 19730530 <--

AB The 2,2-dialkyl-1,3-dihydroquinoxalino[2,3-c]pyrroles I (R, R1 = alkyl) were prepared by treating 2,3-bis(bromomethyl)quinoxaline (II) with amines HNRR1. Thus, 12.7 g II, 5.9 g HNEt2 and alc. were stirred 1 hr at 50° to give I (R = R1 = Et).

IT 40197-25-9P

RN 40197-25-9 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalinium, 2,2-diethyl-2,3-dihydro-, bromide (1:1) (CA INDEX NAME)



• Br-

L9 ANSWER 12 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1975:428279 CAPLUS

DOCUMENT NUMBER: 83:28279

ORIGINAL REFERENCE NO.: 83:4533a,4536a

TITLE: Derivatives of quinoxalino[2,3-c]pyrroline

INVENTOR(S):
Hahn, Witold; Lesiak, Jerzy

PATENT ASSIGNEE(S): Uniwersytet Lodzki

SOURCE: Pol., 2 pp. CODEN: POXXA7

DOCUMENT TYPE: Patent LANGUAGE: Polish

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PL 71049		19740715	PL 1972-155806	19720605 <

GI For diagram(s), see printed CA Issue.

AB The quinoxalinopyrrolines I (R1, R2 = alkyl containing 1-4 C atoms, alkenyl, hydroxyalkyl, arylalkyl, or R1R2 = (CH2)4, CH2CH2OCH2CH2) were obtained in the reaction of the quinoxaline II with secondary aliphatic or heterocyclic amines. Thus, a suspension of 12.7 g II in 100 ml 90% EtOH was treated during 8 hrs at  $30-40^{\circ}$  with a solution of 5.9 g Et2NH in 30 ml EtOH and the whole kept 1 hr at  $50^{\circ}$  to give 76% I (R1 = R2 = Et).

IT 40197-25-9P

RN 40197-25-9 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalinium, 2,2-diethyl-2,3-dihydro-, bromide (1:1) (CA INDEX NAME)

• Br-

L9 ANSWER 13 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1975:156218 CAPLUS

DOCUMENT NUMBER: 82:156218

ORIGINAL REFERENCE NO.: 82:24936h,24937a

TITLE: Di- and tetracyanopyrazines

AUTHOR(S): Rothkopf, Hans W.; Woehrle, Dieter; Mueller,

Reinhardt; Kossmehl, Gerhard

CORPORATE SOURCE: Inst. Org. Chem., Freie Univ. Berlin, Berlin, Fed.

Rep. Ger.

SOURCE: Chemische Berichte (1975), 108(3), 875-86

CASREACT 82:156218

CODEN: CHBEAM; ISSN: 0009-2940

DOCUMENT TYPE: Journal LANGUAGE: German

GI For diagram(s), see printed CA Issue.

AB Diaminomaleonitrile reacts with di- and tetraketones and oxoaldehydes RCOCOR1 (I, R = H, Me, Ph; R1 = H, Me, Ph) to give cyanopyrazines II.

When I is 9,10-phenanthrenequinone, III is formed. Other I, such as 1,8-phenanthroline-9,10-quinone, N-acetylisatin, 4,5:9,10-pyrenediquinone, etc., were also used to give polycyclic II. RC(:NOH)COR1 (R = H, Me; R1 = Ph) could be used instead of I. [HN:C(CN)]2 cyclizes with di- and

tetramines 4,5-RR1C6H2(NH2)2-1,2 to give 2,3-dicyanoquinoxalines IV (R = H, Me, NO2, CO2H; R1 = H, Me), V, and VI. Some dicyanopyrazines cyclize with NH3 to give aminoimino-5H-pyrrolo[3,4-b]pyrazines VII (R = Me, Ph; R1 = H, Me; RR1 = CH:CHCH:CH).

IT 55408-64-5P

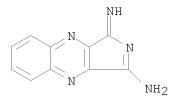
OTHER SOURCE(S):

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of)

RN 55408-64-5 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-3-amine, 1-imino- (CA INDEX NAME)



OS.CITING REF COUNT: 25 THERE ARE 25 CAPLUS RECORDS THAT CITE THIS RECORD (25 CITINGS)

L9 ANSWER 14 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1974:463674 CAPLUS

DOCUMENT NUMBER: 81:63674

ORIGINAL REFERENCE NO.: 81:10149a,10152a

TITLE: 3-Halomethyl-2-quinoxalinecarboxylic acid esters and

their cyclization products with amines

PATENT ASSIGNEE(S): Pfizer Inc.

SOURCE: Brit. Amended, 16 pp.

CODEN: BSXXAH

DOCUMENT TYPE: Patent LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
	GB 1303372		19730117	GB 1970-43229	19700909 <
PRIO	RITY APPLN. INFO.:			US 1970-25543	19700403 <

GI For diagram(s), see printed CA Issue.

AB The title esters I, II, and III were prepared from the appropriate benzofuroxans by successive treatment with MeCOCH2CO2Et and halogenation. Ring closure of I with R2NH2 (R2 = H, Me, Et) gave IV, which are in vitro and in vivo antibacterial agents and animal growth promotants.

IT 40970-22-7P 40970-23-8P 40970-24-9P RL: SPN (Synthetic preparation); PREP (Preparation) (preparation of)

RN 40970-22-7 CAPLUS

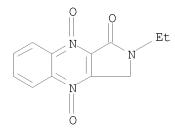
CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 2,3-dihydro-2-methyl-, 4,9-dioxide (CA INDEX NAME)

RN 40970-23-8 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 2,3-dihydro-, 4,9-dioxide (CA INDEX NAME)

RN 40970-24-9 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 2-ethyl-2,3-dihydro-, 4,9-dioxide (CA INDEX NAME)



OS.CITING REF COUNT: 2 THERE ARE 2 CAPLUS RECORDS THAT CITE THIS RECORD

(2 CITINGS)

L9 ANSWER 15 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1974:146108 CAPLUS

DOCUMENT NUMBER: 80:146108

ORIGINAL REFERENCE NO.: 80:23581a,23584a

TITLE: Redox reaction with 2-chloromethylquinoxaline

di-N-oxide

AUTHOR(S): Eholzer, U.; Heitzer, H.; Seng, F.; Ley, K.

CORPORATE SOURCE: Zentralbereich Zent. Forsch.-Wiss. Hauptlab., Bayer

A.-G., Leverkusen, Fed. Rep. Ger.

SOURCE: Synthesis (1974), (4), 296-8

CODEN: SYNTBF; ISSN: 0039-7881

DOCUMENT TYPE: Journal LANGUAGE: German

AB Quinoxaline derivs. I (R = Me, Et, Pr, (CH2)11Me, cyclohexyl, C6H4Cl-p; R1 = cyclohexyl, (CH2)11Me, Bu, Et, Pr, C6H4CO2-H-p) were formed in 31-86% yield by treating the quinoxaline di-N-oxides II with 2 moles R1NH2. I were easily hydrolyzed to the dicarboximides. The pyrroloquinoxalines III (R2 = morpholino, piperidino, pyrrolidino) were obtained in 60-86% yield by treating 2-chloro-methyl-3-cyanoquinoxaline di-N-oxide with 2 moles of the amine.

IT 52398-27-3P 52398-28-4P 52478-80-5P 52478-81-6P 52478-82-7P 52478-83-8P

52478-84-9P 52478-89-4P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of)

RN 52398-27-3 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxaline-1,3(2H)-dione, 2-propyl- (CA INDEX NAME)

RN 52398-28-4 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxaline-1,3(2H)-dione, 2-dodecyl- (CA INDEX NAME)

RN 52478-80-5 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 3-(cyclohexylimino)-2,3-dihydro-2-methyl- (CA INDEX NAME)

RN 52478-81-6 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 3-(dodecylimino)-2-ethyl-2,3-dihydro-(CA INDEX NAME)

RN 52478-82-7 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 3-(cyclohexylimino)-2,3-dihydro-2-propyl- (CA INDEX NAME)

RN 52478-83-8 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 3-(butylimino)-2-dodecyl-2,3-dihydro-(CA INDEX NAME)

RN 52478-84-9 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one,

3-(cyclohexylimino)-2-dodecyl-2,3-dihydro- (CA INDEX NAME)

RN 52478-89-4 CAPLUS

CN Benzoic acid, 4-[(2,3-dihydro-2-methyl-3-oxo-1H-pyrrolo[3,4-b]quinoxalin-1-ylidene)amino]- (CA INDEX NAME)

L9 ANSWER 16 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1973:124629 CAPLUS

DOCUMENT NUMBER: 78:124629

ORIGINAL REFERENCE NO.: 78:20027a,20030a

TITLE: 3-(Halomethyl)-2-quinoxalinecarboxylic acid esters and

their cyclization products with amines

PATENT ASSIGNEE(S): Pfizer Inc.
SOURCE: Brit., 18 pp.
CODEN: BRXXAA

DOCUMENT TYPE: Patent LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.		DATE
GB 1303372	A	19730117	GB 1970-43229		19700909 <
US 3753987	A	19730821	US 1970-25543		19700403 <
CA 969956	A1	19750624	CA 1971-109462		19710402 <
US 3773950	A	19731120	US 1972-235791		19720317 <
PRIORITY APPLN. INFO.:			US 1970-25543	A	19700403 <

GI For diagram(s), see printed CA Issue.

The title quinoxalines (I; R = alkyl; R1 = H, halogen, CF3, Me, MeO; X = C1, Br) and their amine cyclization products (II, R2 = H, alkyl), useful as antibacterials and animal growth promotants, were prepared Thus, addition of benzofuroxan to MeCOCH2CO2Et in NaOEt-EtOH gave I (R = Et; R1 = X = H) which on bromination in DMF with Br2 gave the title compound (I, R = Et, R1 = H, X = Br). Bubbling MeNH2 through the (bromomethyl)quinoxaline in MeCN at 10-13° gave the cyclization product (II, R1 = H, R2 = Me). The same pyrroloquinoxalne was prepared from benzofuroxan and 1-methyl-3-hydroxy-3-pyrrolin-5-one.

IT 40970-22-7P 40970-23-8P 40970-24-9P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of)

RN 40970-22-7 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 2,3-dihydro-2-methyl-, 4,9-dioxide (CA INDEX NAME)

RN 40970-23-8 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 2,3-dihydro-, 4,9-dioxide (CA INDEX NAME)

RN 40970-24-9 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalin-1-one, 2-ethyl-2,3-dihydro-, 4,9-dioxide (CA INDEX NAME)

L9 ANSWER 17 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1973:58357 CAPLUS

DOCUMENT NUMBER: 78:58357

ORIGINAL REFERENCE NO.: 78:9259a,9262a

TITLE: Synthesis of quinoxalino[2,3-c]pyrroline derivatives

AUTHOR(S): Hahn, Witold E.; Lesiak, Jerzy Z. CORPORATE SOURCE: Inst. Chem., Univ. Lodz, Lodz, Pol.

SOURCE: Societatis Scientiarum Lodziensis, Acta Chimica (

1972), 17, 201-5

CODEN: SLACBC; ISSN: 0081-0711

DOCUMENT TYPE: Journal LANGUAGE: Russian

GI For diagram(s), see printed CA Issue.

AB Quinoxalino[2,3-c]pyrrolium bromides (I, R = R1 = Me, Et, Pr, Bu, CH2CH2OH, allyl; R = Me, R1 = PhCH2) and spiroquinoxalino[2,3-c]pyrrolium bromides (II, III, IV) were prepared by treating

2,3-bis(bromomethyl)quinoxaline with secondary amines.

IT 40197-24-8P 40197-25-9P 40197-26-0P 40197-27-1P 40197-28-2P 40197-29-3P

40197-30-6P

RN 40197-24-8 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalinium, 2,3-dihydro-2,2-dimethyl-, bromide (1:1) (CA INDEX NAME)

• Br-

RN 40197-25-9 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalinium, 2,2-diethyl-2,3-dihydro-, bromide (1:1) (CA INDEX NAME)

• Br-

RN 40197-26-0 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalinium, 2,3-dihydro-2,2-dipropyl-, bromide (1:1) (CA INDEX NAME)

RN 40197-27-1 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalinium, 2,2-dibutyl-2,3-dihydro-, bromide (1:1) (CA INDEX NAME)

• Br-

RN 40197-28-2 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalinium, 2,3-dihydro-2,2-bis(2-hydroxyethyl)-, bromide (1:1) (CA INDEX NAME)

• Br-

RN 40197-29-3 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalinium, 2,3-dihydro-2,2-di-2-propen-1-yl-, bromide (1:1) (CA INDEX NAME)

$$\begin{array}{c} \text{CH}_2\text{--}\text{CH} \\ \text{CH}_2\text{--}\text{CH} \\ \text{CH}_2\text{--}\text{CH} \\ \text{CH}_2\text{--}\text{CH} \\ \end{array}$$

• Br-

RN 40197-30-6 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxalinium, 2,3-dihydro-2-methyl-2-(phenylmethyl)-, bromide (1:1) (CA INDEX NAME)

● Br-

OS.CITING REF COUNT: 1 THERE ARE 1 CAPLUS RECORDS THAT CITE THIS RECORD

(1 CITINGS)

L9 ANSWER 18 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1972:461947 CAPLUS

DOCUMENT NUMBER: 77:61947

ORIGINAL REFERENCE NO.: 77:10251a,10254a

TITLE: 3-Arylamino-1-arylpyrrolidine-2,5-diones and their

N-nitroso compounds. II. Properties. New

heterocyclization reaction

AUTHOR(S): Burmistrov. S. I.; Kul'chitskaya, N. E.; Romanneko, V.

D.

CORPORATE SOURCE: Dnepropetr. Khim.-Tekhnol. Inst., Dnepropetrovsk, USSR

SOURCE: Zhurnal Organicheskoi Khimii (1972), 8(5),

1095-100

CODEN: ZORKAE; ISSN: 0514-7492

DOCUMENT TYPE: Journal LANGUAGE: Russian

GI For diagram(s), see printed CA Issue.

AB Cyclodehydration of 11 title nitrosamines (I, R = Ph, substituted Ph; R = Ph, substituted Ph, PhCH2) at 100-20° in Ac2O afforded the corresponding quinoxaline derivs. (II) in 40-60% yield instead of the expected sydnone analogs. Alkaline hydrolysis of II (X = H; R1 = Ph, C6H4Me-p, C6H4OMe-p) gave the corresponding 2,3-quinoxalinedicarboxylic acid mono-N-arylamides (III), which were converted back to II by Ac2O; III were also prepared from 2,3-quinoxalinedicarboxylic anhydride and R1NH2. Similarly, II gave bis-N-arylamides with the resp. R1NH2. Refluxing III in quinoline containing Cu powder yielded 68% 2-quinoxalinecarboxylic acid.

IT 7066-30-0P

RN 7066-30-0 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxaline-1,3(2H)-dione, 2-(phenylmethyl)- (CA INDEX NAME)

L9 ANSWER 19 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1966:456810 CAPLUS

DOCUMENT NUMBER: 65:56810

ORIGINAL REFERENCE NO.: 65:10588g-h,10589a-b

TITLE: N-Benzylimide of quinoxaline-2,3-dicarboxylic acid

AUTHOR(S): Cesari, Adriana

SOURCE: Annali dell'Istituto Superiore di Sanita (1965

), 1(9-10), 555-9

CODEN: AISSAW; ISSN: 0021-2571

DOCUMENT TYPE: Journal LANGUAGE: Italian

GI For diagram(s), see printed CA Issue.

To a boiling suspension of 9.5 g. quinoxaline-2,3-dicarboxylic anhydride AΒ (I) in 300 ml. absolute EtOH, 16 q. PhCH2NH2 (II) was added, the mixture refluxed 1 hr., the solvent evaporated in vacuo, and the residue taken up in H2O and Et2O, to give 12 g. crude benzylamine salt of quinoxaline-2,3-carboxamidic acid (III), m. 182-4° (Me2CO); the crude product was dissolved in warm H2O, the solution filtered, and acidified with HCl, to give 10.5 g. free III, m. 172° (decomposition) (alc.). III (6.5 g.) was treated with 50 cc. SO2Cl2; after 30 min. 300 ml. CHCl3 was added, the mixture refluxed until the solid was completely dissolved, and the solvent evaporated to give 4.8 g. N-benzylimide (IV) of quinoxaline-2,3-dicarboxylic acid, m. 270-2° (C6H6). Pyrolysis of III was accomplished by refluxing the compound in xylene 1 hr. and evaporating the solvent in vacuo, to give a residue of quinoxaline-2-carboxybenzylamide, m. 150-2° (MeOH). Quinoxaline-2,3-dicarboxylic acid monoamide was heated in vacuo to  $\overline{185}^{\circ}$  for 20 min. and to  $\overline{205}^{\circ}$  for another 10 min., to give quinoxaline-2-carboxamide, m. 198° (AcOH). A mixture of 3.9 g. I and 4.2 g. PC15 was gradually heated to  $185^{\circ},$  cooled to  $150^{\circ}$  when the reaction began, kept for 3 hrs. at this temperature, and POC13 was evaporated in vacuo to give 3 g. quinoxaline-2,3-dicarboxylic acid chloride (V), m.  $85-7^{\circ}$  (ligroine). To a solution of 2.5 g. V in 15 ml. anhydrous C6H6, 3 g. II in 50 ml. C6H6 was added slowly, with gentle heating, the mixture was heated 30 min. on a water bath, cooled, the precipitate filtered

and washed with H2O, to give 2.6 g. crude quinoxaline-2,3-dicarboxamide, m.  $190-2^{\circ}$  (alc.). From the mother liquor 0.2 g. IV was isolated by evaporation of the solvent and recrystn. of the residue in C6H6.

IT 7066-30-0P, 2,3-Quinoxalinedicarboximide, N-benzyl-

RL: PREP (Preparation)

(preparation of)

RN 7066-30-0 CAPLUS

off,

CN 1H-Pyrrolo[3,4-b]quinoxaline-1,3(2H)-dione, 2-(phenylmethyl)- (CA INDEX NAME)

L9 ANSWER 20 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1966:456809 CAPLUS

DOCUMENT NUMBER: 65:56809
ORIGINAL REFERENCE NO.: 65:10588e-g

TITLE: Quinoxaline derivatives. IX. An unusual chlorine substitution in quinoxaline N-oxides. Its scope and

limitations

AUTHOR(S): Ahmad, Yusuf; Habib, M. S.; Ziauddin; Bakhtiari,

Bushra

CORPORATE SOURCE: Chem. Res. Div., Pakistan Council Sci. Ind. Res.,

Karachi

SOURCE: Journal of Organic Chemistry (1966), 31(8),

2613-16

CODEN: JOCEAH; ISSN: 0022-3263

DOCUMENT TYPE: Journal LANGUAGE: English

AB cf. CA 64, 5092f. An O function at C-3 in quinoxaline 1-oxides was shown to control the nucleophilic Cl substitution at C-6 observed when these N-oxides are heated with AlCl or ethanolic HCl. In its absence the Cl substitution (a) fails to take place as evidenced in the case of 2,3-diphenylquinoxaline 1-oxide and 1,4-dioxide; (b) if it takes place as in the case of 2,3-dimethylquinoxaline 1-oxide and 1,4-dioxide is directed to the Me groups; (c) takes place at a position adjacent to the N-oxide if it is previously unoccupied. 17 references.

IT 7066-30-0P, 2,3-Quinoxalinedicarboximide, N-benzyl-

RN 7066-30-0 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxaline-1,3(2H)-dione, 2-(phenylmethyl)- (CA INDEX NAME)

OS.CITING REF COUNT: 4 THERE ARE 4 CAPLUS RECORDS THAT CITE THIS RECORD (4 CITINGS)

L9 ANSWER 21 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1962:456273 CAPLUS

DOCUMENT NUMBER: 57:56273

ORIGINAL REFERENCE NO.: 57:11194g-i,11195a-h

TITLE: The catalytic reduction of the imide and anhydride of

quinoxaline-2,3-dicarboxylic acid

AUTHOR(S): Bettinetti, Gian Franco; Tisselli, Eugenio

CORPORATE SOURCE: Univ. Pavia, Pavia, Italy

SOURCE: Annali di Chimica (Rome, Italy) (1961), 51,

1102-12

CODEN: ANCRAI; ISSN: 0003-4592

DOCUMENT TYPE: Journal LANGUAGE: Unavailable

AB To 2 g. of the anhydride of quinoxaline-2,3-dicarboxylic acid (1) in 150 cc. dry tetrahydrofuran (THF), 0.4 g. Pd-C is added, and the suspension reduced at room temperature and pressure. Two atoms H are absorbed to give a blue product, m. 230°; sublimed, it m. 212°. By sapon, in cold dilute NaOH and acidification with HCl, the blue dihydro form (II) of quinoxaline-2,3-dicarboxylic acid is obtained. To 1.99 g. of the imide of quinoxaline-2,3-dicarboxylic acid in 150 cc. dry THF, 0.4 g. Pd-C is added and then reduced at room temperature. It is then evaporated to dryness and a

blue

product is obtained, m. 290° (decomposition). It is sublimed at 10-3 mm. and gives a blue sublimate, m. 280° (decomposition). The imide of 1-acetyl-1,4-dihydroquinoxaline-2,3-dicarboxylic acid (III) is prepared by adding 4 g. I in 50 cc. of HOAc to 0.8 g. Pd-C and the suspension reduced at room temperature and pressure. After hydrogenation, 10 cc. AcCl is added in 3 parts, with stirring well after each addition. The precipitate is filtered

after 24 hrs. to give 5 g. product which is extracted twice with 50 cc. boiling HOAc, to give 3 g. of a red product, m.  $239-40^{\circ}$  (decomposition). By recrystn. from HOAc III is obtained as red crystals, m.  $239^{\circ}$  (browning) and decompose at  $244^{\circ}$ . The N-acetyl imide (VI) of III is prepared by adding 0.8 g. Pd-C to 4 g. I in 50 cc. glacial HOAc and then proceeding as above. When the hydrogenation is completed, 50 cc. Ac2O is added and the mixture heated to the b.p. 30 min. The catalyst is then filtered off and the solution evaporated to dryness in vacuo to give 5.4 g. of

red substance, m. 179-80° (decomposition). It is crystallized from MePh. VI is obtained, which turns brown at 190° and decompose at 202-6°. To 2.41 g. IV in 20 cc. glacial HOAc, 0.5 g. Pd-C is added and the mixture treated as above. After 296 cc. H are absorbed, the reaction is stopped and 5 cc. AcCl is added and the mixture kept 5 days. Then it is heated to 80°, the catalyst filtered off, and the solution evaporated to dryness in vacuo to give 2.5 g. of a red product, decompose at 179-84°. It is recrystd. successively from anhydrous MePh and the product turns brown at 190° and decompose at 202-6°. VI is also prepared by refluxing 2 g. III in 50 cc. Ac20. Evaporation of the solution to

dryness under reduced pressure gives 2.1 g. of a red substance which decompose at 170-80°. Successive recrystns. from anhydrous MePh gives a product which turns brown at 190° and decompose at 202-6°. III (7.2 g.) or 8.5 g. VI was refluxed 2 1/2 hrs. with 50 cc. Ac20. The solution evaporated to dryness trader reduced pressure gave 9 q. of a brown substance, extracted with anhydrous C6H6. The C6H6 exts. were combined and filtered hot. On cooling 6.5 g. of an orange product separated, decompose at 177°. It is crystallized from anhydrous C6H6 and gives the N-acetylimide of 1,4-diacetyl-1,4-dihydroquinoxaline-2,3-dicarboxylic acid (VII), decompose at  $180^{\circ}$ . To 1 g. VI suspended in 15 cc. H2O, 10 cc. concentrated NH3 is added with stirring. After a short time 0.7 g. of a crystalline substance was precipitated, filtered off, and dried. The product turns red at 135° and m. 172° (decomposition). After crystallization from H2O it turns red and m. at 173  $^{\circ}$  (decomposition), giving the diamide of 1-acetyl-1, 4-dihydroqulnoxaline-2, 3-dicarboxylic acid (VIII). VI (0.5 g.) is dissolved in 5 co. cold 5% NaHCO3. The solution is filtered through C and acidified with dilute HCl to give 0.48 g. of a yellow product, m. 168-70°. By recrystn. from THF, the N-acetylmonoamide (IX) of 1-acetyl-1, 4-dihydroquinoxaline-2, 3-dicarboxylic acid is obtained, m. 172°. IX (0.5 g.) is boiled 2 1/2 hrs. in 5 cc. Ac20, and then the excess A2O removed. Crystallization of the residue from C2H6, gave 0.35 g. of

the

yellow triacetyl derivative To 3.27 g. VII in 50 cc. glacial HOAc is added 0.35 g. Pd-C and reduced as above. After 14-15 hrs. the hydrogenation is stopped (259 cc. H are absorbed). The catalyst is illtered off, 5 cc. Ac20 added, and concentrated under reduced pressure to a paste. The mass is kneaded in 30 cc. dry Et2O and filtered off to give 2.6 g. of a product which is crystallized from Ac20 to give the N-acetylimide (X) of 1,4-diacetyl-1,2,3,4-tetrahydroquinoxaline-2,3-dicarboxylic acid, m.  $220-3^{\circ}$  (decomposition). X (0.5 g.) was dissolved in 5 cc. boiling H2O, the solution filtered and cooled to give 0.2 g. product, m.  $290-1^{\circ}$ (decomposition), not changed on recrystn., the N-acetylimide (XI) or 1-acetyl-1,2,3,4-tetrahydroquinoxaline-2,3-dicarboxylic acid as shown by the infrared spectrum. X (2.4 g.) and 15 cc. 10% NaOH is boiled 2 1/2 hrs. until no more NH3 is evolved. The solution is cooled, acidified with concentrated HCl, and filtered after 12 hrs. to give 1.47 g. product, m.  $219-20^{\circ}$ . After remaining 48 hrs. in the mother liquor 0.2 g. of a second product separated, m. 180° (decomposition). The product, m. 219-20° is crystallized from H2O, m. 219-20°. 1 - Acetyl -1,2,3,4 - tetrahydroquinoxaline -2,3 - dicarboxylic acid (XII) (1.1 g.) is recovered. The product, m.  $180^{\circ}$ , after recrystn. from H2O decompose at  $213-16^{\circ}$ . With this information and the infrared spectrum it was

shown to be 1,2,3,4-tetrahydroquinoxaline-2,3-dicarboxylic acid. XII (0.3 g.) is refluxed in 3 cc. Ac20  $15 \, \mathrm{min}$ . On cooling 0.24 g. of a product crystallized, m. 220° (decomposition). After recrystn. from Ac20 it decomposed at  $226-8^{\circ}$  with browning at  $205-15^{\circ}$ . From this data and the infrared spectrum it was shown to be the anhydride of N, N'-diacetyl-1, 2, 3, 4-tetrahydroquinoxaline-2, 3-dicarhoxylic acid. ΙT 96954-22-2P, 2,3-Quinoxalinedicarboximide, N,1-diacetyl-1,4-dihydro-97159-76-7P, 2,3-Quinoxalinedicarboximide, N,1-diacetyl-1,2,3,4-tetrahydro-97470-26-3P, 2,3-Quinoxalinedicarboximide, N, 1, 4-triacetyl-1, 4-dihydro- 97724-78-2P, 2,3-Quinoxalinedicarboximide, N,1,4-triacetyl-1,2,3,4-tetrahydro-856949-70-7P, 2,3-Quinoxalinedicarboximide, 1-acetyl-1,4-dihydro-RL: PREP (Preparation) (preparation of) RN 96954-22-2 CAPLUS 1H-Pyrrolo[3,4-b]quinoxaline-1,3(2H)-dione, 2,4-diacetyl-4,9-dihydro- (CA CN INDEX NAME)

RN 97159-76-7 CAPLUS CN 1H-Pyrrolo[3,4-b]quinoxaline-1,3(2H)-dione, 2,4-diacetyl-3a,4,9,9a-tetrahydro- (CA INDEX NAME)

RN 97470-26-3 CAPLUS
CN 1H-Pyrrolo[3,4-b]quinoxaline-1,3(2H)-dione, 2,4,9-triacetyl-4,9-dihydro(CA INDEX NAME)

RN 97724-78-2 CAPLUS
CN 1H-Pyrrolo[3,4-b]quinoxaline-1,3(2H)-dione,
2,4,9-triacetyl-3a,4,9,9a-tetrahydro- (CA INDEX NAME)

RN 856949-70-7 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxaline-1,3(2H)-dione, 4-acetyl-4,9-dihydro- (CA INDEX NAME)

IT 5660-33-3, 2,3-Quinoxalinedicarboximide

(reduction of)

RN 5660-33-3 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxaline-1,3(2H)-dione (CA INDEX NAME)

L9 ANSWER 22 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1962:456272 CAPLUS

DOCUMENT NUMBER: 57:56272
ORIGINAL REFERENCE NO.: 57:11194c-g

TITLE: Potential amebicides. XIII. Synthesis of Mannich bases

and iodo derivatives of some 3-alkyl-8-hydroxy-4-quinazolones

AUTHOR(S): Iyer, R. N.; Dhar, M. L.

CORPORATE SOURCE: Central Drug Research Inst., Lucknow

SOURCE: Journal of Scientific & Industrial Research (

1961), 20C, 175-7

CODEN: JSIRAC; ISSN: 0022-4456

DOCUMENT TYPE: Journal LANGUAGE: Unavailable

GI For diagram(s), see printed CA Issue.

AB cf. CA 55, 27206e; 57, 11159e. Syntheses of the title compds. were described. 3-Ethyl-8-hydroxy-4-quinazolone (0.95 g.) in 5 ml. 10% HCl added to a cooled solution of 10 ml. iodine monochloride, the precipitate extracted with

 ${\tt C6H6},$  the extract washed with  ${\tt H2O}$  and aqueous  ${\tt Na2S2O3}$  and dried, and the solvent

removed in vacuo gave 7-iodo-3-R-8-hydroxy-4-quinazolone (I) (R = Et), m. 182  $^{\circ}$  (EtOH). Similarly were prepared I (R = Pr), m. 160 $^{\circ}$ , and I (R = Bu), m. 150°. The Mannich bases were prepared as in the following example: piperidine (0.5 ml.) added to a cooled solution of 0.9 g. 3-methyl-8-hydroxy-4-quinazolone in 50 ml. EtOH, the mixture kept 1 hr. at room temperature and then refluxed 6 hrs., EtOH distilled, the residue dissolved in C6H6, the solution dried, and the solvent removed gave 7-piperidinomethyl-2-R-3R'-8-hydroxy-4-quinazolone (II) (R = H, R' = Me), m. 152° (C6H6-petr. ether). The following II were prepared (R, R', and m.p. given): H, Et, 134°; H, Pr, 131° H, Bu, 121°; Me, Me, 125°; Me, Et, 91°; Me, Pr, 104°. The following 7-morpholinomethyl analogs of II were prepared (R, R', and m.p. given): H, Me, 164°; H, Et, 128°; H, Pr, 86°; H, Bu, 111°; Me, Me, 153°; Me, Et, 97°; Me, Pr, 106°. The following III were prepared (R, R', and m.p. given): H, Me, 259°; H, Et, 215°; H, Pr, 230° Me, Me, 5660-33-3, 2,3-Quinoxalinedicarboximide ΙT (reduction of) RN 5660-33-3 CAPLUS CN 1H-Pyrrolo[3,4-b]quinoxaline-1,3(2H)-dione (CA INDEX NAME)

OS.CITING REF COUNT: 1 THERE ARE 1 CAPLUS RECORDS THAT CITE THIS RECORD (1 CITINGS)

L9 ANSWER 23 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1959:45125 CAPLUS

DOCUMENT NUMBER: 53:45125

ORIGINAL REFERENCE NO.: 53:8103a-i,8104a-c

TITLE: Spectral study of cyclic ketones. III. Pyrrolidine

series

AUTHOR(S): Sandris, Constantin; Ourisson, Guy

CORPORATE SOURCE: Inst. Chem., Strasbourg

SOURCE: Bulletin de la Societe Chimique de France (

1958) 345-9

CODEN: BSCFAS; ISSN: 0037-8968

DOCUMENT TYPE: Journal Unavailable OTHER SOURCE(S): CASREACT 53:45125

AB Phorone (100 g.) stirred 3 days with 320 ml. NH3 (d. 0.904), the solution
warmed 0.5 hr. on the steam bath to 80°, cooled, and saturated with
Na2CO3 gave 85 g. triacetoneamine (I), m. 55-60°, b14 84°
which was sublimed in vacuo, m. 37-9° (needles), λ 292 (18),
298 (18), v 3530 (NH), 1708 (CO); hydrate, m. 58-60° (platelets
from Et2O-H2O); hydrobromide, m. 203° (needles, EtOH-Et2O),
λ 292 (15), v 1730 (CO); acetate, m. 100-2° (from the
hydrate of I with Ac2O). I (2.85 g.) heated 1 hr. on the steam bath with
4.5 ml. Ac2O gave 1.2 g. N-acetyl-triacetoneamine (II), m. 62-4°,
v 1724 (CO), 1634 (CO, amide); 2,4-dinitrophenylhydrazone, m.
170° (decomposition, yellow needles, C6H6). Br (76 g.) in 100 ml. AcOH
added dropwise with stirring to 37 g. I in 150 ml. AcOH gave 74 g.
α,α'-dibromotriacetoneamine hydrobromide (III), m.

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193-5° (decomposition, EtOH), \lambda 300 (241), \nu 1760 (CO). III
     stirred with 750 ml. NH3 and the solution saturated with Na2CO3 gave 50 g.
     3,3,5,5-tetramethyl-4-azacyclopent-1-ene-1-carboxamide (IV), m.
     178-9^{\circ} (needles, C6H6 then sublimed), \lambda 210 (9900). IV (30
     g.) in 250 ml. H2O added to an ice-cold solution of 35 g. Br in 150 ml. H2O
     containing 43 g. Na2CO3, the mixture heated 1 hr. on the steam bath, cooled,
200
     q. Na2CO3 added, and steam distilled, the distillate collected in aqueous HCl,
     the acidic solution evaporated under reduced pressure, 100 ml. H2O added,
     in ice, and 200 ml. 50% KOH added gave 10.5 g. brown oil which was
     chromatographed in Et2O solution on Al2O3 to give
     2,2,4,4-tetramethyl-3-azacyclopentanone (V), b747 169°, n24D 1.446,
     \lambda 301 (37), \lambda (EtOH-HClO4) 292 (20), \nu 3360 (NH), 1750
     (CO), pKa 6.1 (80% methylcellosolve), 6.6 (50% EtOH), 7.1 (H.20);
     benzylidene derivative m. 89-91° (yellow needles, EtOH-H2O, then
     sublimed), \lambda 230 (7700), 294 (1300), \lambda (EtOH-HClO4) 231
     (8200), 304 (13,200), v 3380 (NH), 1720 (CO), pKa 5.2 (80%
     methylcellosolve), 5.9 (50% EtOH); perchlorate of benzylidine derivative m.
     214-16^{\circ} (decomposition, H2O). V (1 g.) heated on the steam bath with 1
     ml. Br, the mixture cooled, diluted with Et2O, and Na2CO3 solution added gave
1.3
     q. 5,5-dibromo-2,2,4,4-tetramethyl-3-azacyclopentanone, m. 35-7°,
     \lambda 318 (41), \lambda (EtOH-HClO4) 306 (92), \nu 3356 (NH), 1776
     (CO). V (1.85 g.) stirred 0.5 hr. under N with 1 g. K in 40 ml.
     tert-BuOH, 2.7 g. isoamyl nitrite added, stirred overnight, decomposed with
     {
m H2O}, vacuum-distilled under {
m CO2}, and the aqueous concentrate adjusted to pH 7.4
     with a current of CO2 gave 1.6 g. 2-hydroxyamino-3,3,5,5-tetramethyl-4-
     azacyclopentanone (VI) which was separated into 2 isomers, (VIa) (about 10% of
     total), m. 141-2^{\circ} (decomposition in sealed tube), insol. in C6H6,
     \lambda 264 (15,700), and (VIb) (about 90%), m. 142-5° (decomposition
     in sealed tube), soluble in C6H6, \lambda 248 (7600). VIb is assigned the
     chelated cis structure. VI (500 mg.) added to 3.3 g. NaHSO3 in 25 ml. H2O
     saturated with SO2, allowed to stand overnight, heated 3 hrs. on the steam
     bath, cooled, saturated with SO2, decomposed with Na2CO3 solution saturated
with NaCl,
     and extracted with CHCl3 followed by continuous extraction with Et20 gave 355
ma.
     3,3,5,5-tetramethyl-4-azacyclopentane-1,2-dione (VII), m. 113-16°
     (red needles), \lambda 295 (53, s), 318 (61), \lambda (EtOH-HClO4) 292
     (66), 308 (68), v 3344 (NH), 1749 (CO); hydrate m. 110-14°
     (becomes red about 95°), \lambda 304 (54), 318 (57), \lambda
     (EtOH-HClO4) 290 (69), 304 (65), v 3484 (OH), 3322 (NH), 1751 (CO).
     VII refluxed 1 hr. in EtOH with o-(H2N)2C6H3 gave the quinoxaline, m.
     118-20^{\circ}, \lambda 239 (26,200), 310 (7200, s), 322 (9600). V (2
     g.) heated 1 hr. on the steam bath with 3.1 g. Ac20, H2O added, the mixture
     neutralized with NaHCO3, and extracted with Et2O gave 2.3 g.
     2,2,4,4-tetramethyl-3-acetyl-3-azacyclopentanone (VIII), m. 73-4°,
     \lambda 286 (31), 292 (30) (inflection), \lambda unchanged in
     EtOH-HClO4, \nu 1773 (CO), 1640 (CO amide); benzylidene derivative m.
     139.5-40.5° (dilute EtOH, then sublimed), \lambda 232 (9200), 305
     (14,300), \lambda unchanged in EtOH-HClO4, \nu 1721 (CO), 1647 (CO
     amide), 1613 (C:C). VIII was not hydrolyzed by 20% HCl, 85% H3PO4, or 10%
     NaOH, all at 100°. VIII (850 mg.) refluxed 1 hr. with 770 mg. SeO2
     in 9 ml. AcOH, the Se filtered off, the filtrate neutralized with NaHCO3,
     saturated with NaCl, extracted with Et20, and treated with Hg gave 750 mg.
     3,3,5,5-tetramethyl-4-acetyl-4-azacyclopentane-1,2-dione (IX), m.
     120-2^{\circ} (yellow-orange needles, sublimed), \lambda 313 (59),
     \lambda unchanged in EtOH-HClO4, \nu 1764 (CO), 1639 (CO amide);
     hydrate, m. 93-6° (colorless, m. to an orange liquid), \lambda 308
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(41),  $\nu$  3510, 3390 (OH), 1779 (CO), 1610 (CO amide). IX with o-(H2N)2C6H4 gave the quinoxaline derivative, m. 212-14°,

 $\lambda$  238 (32,700), 310 (7700, s), 323 (10,900). IX (300 mg.) refluxed 1 hr. with 1 g. KOH in 5 ml. H2O, the mixture cooled, diluted with H2O, acidified with H2SO4, saturated with NaCl, and extracted with Et2O gave 210 mg. 1-hydroxy-2,2,4,4-tetramethyl-3-acetyl-3-azacyclobutane-1-carboxylic acid (X), m.  $205-10^{\circ}$  (decomposition), v 3460 (OH), 1684 (acid); Me ester m.  $119-21^{\circ}$  (sublimed) v 1742 (CO ester), 1597 (CO amide). X (55 mg.) in 2 ml. CHC13 refluxed with 150 mg. Pb(OAc)4 in 2 ml. CHC13 gave impure 2, 2, 4, 4-tetramethyl-3-acetyl-3-azacyclobutanone. V (1.5 g.) and 1.9 q. BzCl in 15 ml. C5H5N overnight gave an oil which was chromatographed on Al203 (1:1 petr. ether-Et20) to give 1.9 g. 2,2,4,4-tetramethyl-3-benzoyl-3-azacydopentanone (XI), m.  $50-2^{\circ}$ , no maximum in ultraviolet, v 1757 (CO), 1626 (CO amide), 1603 (C:C); benzylidene derivative m. 124-6° (dilute EtOH),  $\lambda$  232 (11,500, s), 312 (16,100),  $\lambda$  unchanged in EtOH-HClO4,  $\nu$  1709 (CO), 1626 (CO amide), 1680 (C:C.). XI (570 mg.) refluxed 1 hr. with 387 mg. SeO2 in 10 ml. AcOH gave 3,3,5,5-tetramethyl-4-benzoyl-4-azacyclopentane-1,2-dione, m. 107-11°,  $\lambda$  311 (70),  $\nu$  1783, 1770 (CO), 1643 (CO amide), 1608 (C:C); hydrate m. 97-100° (colorless, melts to a yellow liquid),  $\nu$  3509, 3175 (OH), 1779 (CO), 1600 (CO amide). 108875-28-1P, 2H-Pyrrolo[3,4-b]quinoxaline, ΤT 1,3-dihydro-1,1,3,3-tetramethyl- 133231-22-8P, 2H-Pyrrolo[3,4-b]quinoxaline, 2-acetyl-1,3-dihydro-1,1,3,3-tetramethyl-RL: PREP (Preparation) (preparation of) 108875-28-1 CAPLUS RN 1H-Pyrrolo[3,4-b]quinoxaline, 2,3-dihydro-1,1,3,3-tetramethyl- (CA INDEX CN

RN 133231-22-8 CAPLUS

CN Ethanone, 1-(1,3-dihydro-1,1,3,3-tetramethyl-2H-pyrrolo[3,4-b]quinoxalin-2-yl)- (CA INDEX NAME)

OS.CITING REF COUNT: 6 THERE ARE 6 CAPLUS RECORDS THAT CITE THIS RECORD (6 CITINGS)

L9 ANSWER 24 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1959:45124 CAPLUS

DOCUMENT NUMBER: 53:45124

ORIGINAL REFERENCE NO.: 53:8102f-i,8103a

TITLE: Spectral study of cyclic ketones. II. Hydration of

non-enolizable lpha-diketones

AUTHOR(S): Sandris, Constantin; Ourisson, Guy

CORPORATE SOURCE: Inst. Chem., Strasbourg

Bulletin de la Societe Chimique de France ( SOURCE:

1958) 338-44

CODEN: BSCFAS; ISSN: 0037-8968

DOCUMENT TYPE: Journal LANGUAGE: Unavailable GΙ For diagram(s), see printed CA Issue.

AΒ

cf. C.A. 50, 13608c. [ $\lambda$  in m $\mu$ , ( $\epsilon$ ), taken in EtOH unless stated otherwise, shoulder = s; v from Nujol mull in cm.-1] Anhydrous 3,3,5,5-tetramethyl-4-oxa-1,2-cyclopentanedione (I) red needles, m. 55°, when exposed to damp air or by evaporation of its aqueous solution gives a dihydrate (II), colorless crystals, m. 71-3° (Et20-petr. ether),  $\lambda$  319 (42),  $\nu$  3250, 3540 (OH), 1773 (CO), 1642 (H2O of crystallization), and a hemihydrate (III), white crystals, m. 118-19° to a red liquid  $\lambda$  325 (44), unchanged in EtOH-HCl,  $\nu$  3430 (OH), 1765 (CO). III (500 mg.) in 10 ml. oxygenated H2O added to 200 mg. Na2CO3 in 5 ml. H2O, the mixture acidified and extracted with Et20 gave (HO2CCMe2)20, m. 153-5°. III gave no Me ether with MeOH-HCl. III gave an increase in acidity in H3BO3 solution, II gave no increase. III (330 mg.), 0.22 ml. SOC12, and 0.48 ml. C5H5N refluxed 1 hr. in 20 ml. C6H6 gave a cyclic sulfite, m. 120-2°,  $\lambda$  329 (55),  $\nu$  1786 (CO), 1231 (SO). Structures are proposed for II (HO)2C.CO.CMe2.O.CMe2.H2O and III. DL-Oxocineole (IV) m.  $41-2^{\circ}$ ,  $\lambda$  302 (46),  $\nu$  1745 (CO); benzilidene derivative m. 90-2°,  $\lambda$  224 (7950), 228 (7500), 294 (18,100), v 1715 (CO), 1631 (C:C). IV heated 1 hr. with SeO2 in AcOH gave dioxocineol, m. 65-8 $^{\circ}$  (yellow crystals),  $\lambda$  310 (51), v 1751 (CO); hydrate, m. 80-4 $^{\circ}$  (colorless, melts to a yellow

ΙT 108875-28-1 133231-22-8

(Derived from data in the 6th Collective Formula Index (1957-1961))

108875-28-1 CAPLUS RN

1H-Pyrrolo[3,4-b]quinoxaline, 2,3-dihydro-1,1,3,3-tetramethyl- (CA INDEX CN NAME)

liquid),  $\lambda$  313 (45),  $\nu$  3344, 3257-3195 (OH), 1745 (CO). The

hydration of other diketones is discussed.

RN 133231-22-8 CAPLUS

Ethanone, 1-(1,3-dihydro-1,1,3,3-tetramethyl-2H-pyrrolo[3,4-b]quinoxalin-2-CN yl) - (CA INDEX NAME)

THERE ARE 3 CAPLUS RECORDS THAT CITE THIS RECORD OS.CITING REF COUNT: 3 (3 CITINGS)

L9 ANSWER 25 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1959:45123 CAPLUS

DOCUMENT NUMBER: 53:45123 ORIGINAL REFERENCE NO.: 53:8102f

TITLE: Chemistry of naturally occurring furans

AUTHOR(S): Levisalles, I. J.

CORPORATE SOURCE: Imp. Coll. Sci. and Technol., London

SOURCE: Perfumery and Essential Oil Record (1958),

49, 504-11

133231-22-8

CODEN: PEORAA; ISSN: 0369-8998

DOCUMENT TYPE: Journal LANGUAGE: Unavailable AB A review with 49 references.

(Derived from data in the 6th Collective Formula Index (1957-1961))

RN 108875-28-1 CAPLUS

108875-28-1

CN 1H-Pyrrolo[3,4-b]quinoxaline, 2,3-dihydro-1,1,3,3-tetramethyl- (CA INDEX

NAME)

ΙT

RN 133231-22-8 CAPLUS

CN Ethanone, 1-(1,3-dihydro-1,1,3,3-tetramethyl-2H-pyrrolo[3,4-b]quinoxalin-2-yl)- (CA INDEX NAME)

L9 ANSWER 26 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1959:45122 CAPLUS

DOCUMENT NUMBER: 53:45122
ORIGINAL REFERENCE NO.: 53:8102c-f

TITLE: Synthesis of novel oxides in the series of

5,5-dimethyl-decahydronaphthalene

AUTHOR(S): Mousseron, Max; Mousseron-Canet, Magdeleine; Granier,

Marcel

CORPORATE SOURCE: Ecole Nat'l. Sup. Chim., Montpellier, Fr.

SOURCE: Comp. rend. (1958), 247, 564-8

DOCUMENT TYPE: Journal LANGUAGE: Unavailable CASREACT 53:45122 AB cf. Diels, et al., C.A. 23, 3692.

4-(4-Methyl-3-pentenyl)-4-cyclohexene-1,2-dicarboxylic acid (I) treated

with 98% HCO2H (II) containing some H2SO4 yielded

 ${\tt decahydro-8a-hydroxy-8,8-dimethylnaphthalenedicarboxylic acid}$ 

 $\gamma$ -lactone (III), m. 206-7°.

4,5-Dimethyl-4-cyclohexene-1,2-dicarboxylic acid, m. 197°, with II vielded 5-hydroxy-4,5-dimethyl-cyclohexane-1,2-dicarboxylic acid γ-lactone, m. 154°. The acid anhydride of I with II at 70° yielded 8,8-dimethylheptahydro-2,3-naphthalenedicarboxylic anhydride (IV), m. 98-100°, b0.5 150°. IV was hydrolyzed to the corresponding diacid (V), m. 170°. V treated with II at 70° gave III. I with CH2N2 gave the corresponding di-Me ester, which with LiAlH4 yielded 4-(4-methyl-3-pentenyl)-4-cyclohexene-1,2-dimethylol (VI), b0.4 160-5°. VI was dehydrated to 3a,4,7,7a-tetrahydro-5-(4-methyl-3-pentenyl)phthalan, b0.5 110-12°, cyclized by II at 70° to 1,3,3a,4,5,6,7,8,9,9a-decahydro-5,5-dimethylnaphtho[2,3-c]furan, b0.7 100-2°. The 3a,5,5-tri-Me derivative, b0.5 98-9°, was obtained similarly.

IT 108875-28-1 133231-22-8

(Derived from data in the 6th Collective Formula Index (1957-1961))

RN 108875-28-1 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxaline, 2,3-dihydro-1,1,3,3-tetramethyl- (CA INDEX NAME)

RN 133231-22-8 CAPLUS

CN Ethanone, 1-(1,3-dihydro-1,1,3,3-tetramethyl-2H-pyrrolo[3,4-b]quinoxalin-2-yl)- (CA INDEX NAME)

L9 ANSWER 27 OF 28 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1959:34827 CAPLUS

DOCUMENT NUMBER: 53:34827

ORIGINAL REFERENCE NO.: 53:6240i,6241a-i,6242a-i,6243a

TITLE: Condensation of bis(halomethyl) compounds with

non-aromatic amines

AUTHOR(S): Ried, Walter; Grabosch, Joachim

CORPORATE SOURCE: Univ. Frankfurt, Germany

SOURCE: Chemische Berichte (1958), 91, 2485-95

CODEN: CHBEAM; ISSN: 0009-2940

DOCUMENT TYPE: Journal Unavailable OTHER SOURCE(S): CASREACT 53:34827

AB Isocyclic and heterocyclic compds. with 2 ortho- or perihalomethyl groups yield at room temperature with primary aliphatic amines cyclic tertiary amines and with secondary aliphatic amines cyclic quaternary ammonium salts.

2,3-Bis(chloromethyl)thianaphthene (I) (14.1 g.) and 12.5 g. KOAc in 250 cc. glacial AcOH refluxed 4.5 hrs., filtered hot, evaporated in vacuo, the

crystalline residue shaken with 150 cc. C6H6 and 150 cc. H2O, and the C6H6 layer evaporated gave 15-16 g. oily 2,3-bis(acetoxymethyl)thianaphthene (II). II (9 g.), 20 g. KOH, 80 cc. H2O, and 40 cc. EtOH refluxed 3 hrs., cooled, extracted with Et2O, and the extract evaporated yielded 5.4-5.9 g. 2,3-bis(hydroxymethyl)thianaphthene (III), needles, m. 136-7° (C6H6). III (3.4 g.) in 250 cc. refluxing Et2O treated during 25 min. with 8 cc. PBr3 in 100 cc. Et2O, refluxed 0.5 hr., poured onto ice, and extracted with Et2O yielded 5.6 g. 2,3-bis(bromomethyl)thianaphthene (IV), pale yellow crystals. Paraformaldehyde (45 g.) in 200 cc. glacial AcOH treated 4-5 hrs. with stirring with dry HBr, the solution treated with 50 g. molten thianaphthene, the mixture heated 15 min. at 60°, cooled, stirred 1-2 hrs., filtered, the crude product dissolved in 200 cc. hot C6H6, the solution diluted with 200 cc. petr. ether, boiled with C, filtered, and cooled to 0° deposited 50.5 g. IV, m. 138-9°, which was also obtained from thianaphthene with the equivalent amount of (BrCH2)2O at

room

temperature The appropriate bis(halomethyl) compound (1 mole) [or 0.5 mole tetrakis(halomethyl) compound] in dry C6H6 treated with 3 moles primary amine in dry C6H6, kept at room temperature, filtered, and the filtrate worked up gave the corresponding condensation product. BuNH2 (3.4 g.) and 4.4 g. 1,8-C10H6(CH2Br)2 (V) in 150 cc. dry C6H6 gave during 1 day a light yellow, mobile oil; a 2.45-g. portion digested with 15 cc. dilute HCl, heated briefly to  $60^{\circ}$ , and cooled to  $0^{\circ}$  yielded 1.63 g. 2-butyl-2-azaperinaphthindan-HCl (VI.HCl), m. 238-41° (decomposition) (Me2CO). Crude VI in Et2O with excess MeI gave VI.MeI, m. 196-8°. V (3.17 g.), 3.3 g. PhCH2NH2, and 85 cc. C6H6 yielded during 10 hrs. 2.7 g. crude product; a 2.62-g. portion dissolved on the steam bath in 280 cc. dilute HCl and cooled gave 2.9 g. 2-PhCH2 analog (VII) of VI.HCl, hygroscopic, leaflets, m. 252.5-4.5° (absolute EtOH). V (5.0 g.), 4.8 g. cyclohexylamine (VIII), and 100 cc. C6H6 yielded during 4-5 hrs. the 2-cyclohexyl analog (IX) of VI, leaflets, m. 71-2° (aqueous EtOH); picrate, needles, m. 186-7° (decomposition) (60% EtOH). Crude IX (3.9 g.) treated with 20 cc. warm dilute HCl and the crude product recrystd. from 50% EtOH gave IX.HCl, leaflets, m. 295° (decomposition). IX (0.69 g.) in 20 cc. Et20 with 0.3 cc. MeI yielded 0.60 g. IX.MeI, light gray leaflets, m. 260-1° (EtOH). 1,2-C10H6(CH2Br)2 (X) (5.0 g.), 5.2 g. PhCH2NH2, and 180 cc. C6H6 allowed to stand overnight, the resulting oily deposit boiled 3 times with 40 cc. each and once with 80 cc. dilute HCl, and the combined hot exts. cooled deposited 1.1 q. 1,2-C10H6(CH2NHCH2Ph)2.2HCl.0.5H2O, m. 257-8° (BuOH or absolute EtOH). X (4.0 g.), 3.9 g. VIII, and 160 cc. C6H6 kept 6 hrs., the precipitated yellow, viscous oil refluxed 1 hr. with 60 cc. petr. ether, the extract filtered, concentrated, and cooled to  $-20^{\circ}$  yielded 2-cyclohexyl-1,3-dihydroxybenzo[e]isoindole (XI). Crude XI in EtOH treated with alc. picric acid and allowed to stand several days gave the picrate of XI, needles, m.  $193-4^{\circ}$  (decomposition) (absolute EtOH). XI (1.0 q.) shaken with 40 cc. boiling dilute HCl and 20 cc. H2O and filtered hot gave from the filtrate 0.55 g. XI.HCl, m.  $280-4^{\circ}$  (absolute EtOH). 1,2,4,5-C6H2(CH2Cl)4 (XII) (3.0 g.), 5.0 g. PrNH2, and 200 cc. C6H6 gave during 3 days 2,5-dipropyl-1,3,4,6-tetrahydrobenzo[1,2-c;4,5-c']dipyrrole (XIII), yellow crystals. XIII in EtOH with alc. picric acid gave the dipicrate of XIII, yellow leaflets, m. 219° (decomposition) (BuOH). XII (5.0 g.), 8.9 g. BuNH2, and 150 cc. C6H6 kept 3 days at  $0^{\circ}$ , filtered, washed twice with 100 cc. H2O each, and worked up gave 3.1 g. 2,5-di-Bu analog of XIII, leaflets, m. 121° (Me2CO); dipicrate, greenish yellow powder, m. 211-12° (decomposition) (glacial AcOH). XII (6.0 g.) and 14.8 g. PhCH2NH2 in 400 cc. C6H6 kept 10-12 days, filtered,evaporated, the residue digested with cold ligroine, and recrystd. from EtOH yielded 1.6 g. 2,5-di-PhCH2 analog of XIII, needles, m. 159-61° (MeOH); a portion in CHCl3 treated with alc. picric acid gave the dipicrate, needles which flash on heating without melting. XII (3.0 g.), 6.6 g. VIII, and 200 cc. C6H6 kept 8 hrs., the crude product boiled 0.5

hr. with Et20, and the residue (2.1 g.) recrystd. from absolute EtOH yielded 2,5-dicyclohexyl analog of XIII, plates m.  $238-40^{\circ}$ , yellow in concentrated H2SO4 and glacial AcOH. 2,3-Bis(bromomethyl)quinoxaline (XIV) (3 g.), 2 g. PrNH2, and 190 cc. C6H6 gave during 5 hrs. a crude oily base; the oil shaken with Et20 and H20 and the Et20 layer evaporated gave 0.05 g. 2-propyl-1, 3-dihydropyrrolo[3, 4-b]quinoxaline (XV), light brown prisms, m.  $160-1^{\circ}$  (EtOH). XIV (5 q.) and 4.5 q. BuNH2 kept 24 hrs. at 0° in 160 cc. C6H6, the crude product dissolved in Et2O, the solution diluted with an equal volume of EtOH, and allowed to evaporate slowly during several weeks yielded 7-8% 2-Bu analog of XV, pale yellow needles from EtOH or prisms from petr. ether, m. 118-19°. XIV (20 g.), 20.6 g. PhCH2 NH2, and 530 cc. C6H6 allowed to stand 3.5 hrs., the crude crystalline product boiled with 200 cc. EtOH, and the insol. material recrystd. from BuOH yielded 3.4 g. 2-PhCH2 analog of XV, needles, m. 212.5-14.5°. XIV (5.0 g.) and 4.8 g. VIII in 160 cc. C6H6 allowed to stand 2 hrs., the resinous precipitate digested with Et20 and filtered, the filtrate evaporated,

the

residue boiled with C in 100 cc. EtOH, concentrated to 50 cc., and cooled to -20° yielded the 2-cyclohexyl analog of XV, hygroscopic needles, m. 233-5°. XIV (5.0 g.), 3.7 g. CH2:CHCH2NH2, and 160 cc. C6H6 kept 24 hrs. at 0°, the crude, brown oily product treated with Et2O, and the crystalline material (1.0-1.2 g.) recrystd. from EtOH yielded the 2-allyl analog (XVI) of XV, hygroscopic needles, m. 172°, dissolves in dilute HCl with green color which changed soon to regal blue and during 1-2 hrs. to colorless, dissolves slowly with yellow and olive-green color in concentrated

H2SO4; picrate, needles, m.  $177^{\circ}$  (dioxane). XVI (0.44 g.) in 50 cc. EtOAc added to 0.2 g. Pd-C in 25 cc. EtOAc and hydrogenated 5 hrs. at 15°/760 mm. yielded 2,3-dimethylquinoxaline, m. 104-5° (H2O). I (3 g.), 3 g. PrNH2, and 200 cc. MeOH allowed to stand 2-3 weeks and filtered, the filtrate evaporated, and the residue shaken with Et20 left 1.6 g. 2,3-bis(propylaminomethyl)thianaphthene-2HCl, pale yellow, m. 297-8° (BuOH). I (4.0 g.), 5.2 g. VIII, and 200 cc. C6H6 kept 6 weeks at room temperature and the oily product treated with Et20 or Me2CO gave solid 2-cyclohexyl-1,3-dihydropyrrolo[3,4-b]thianaphthene (XVII). Crude XVII boiled briefly with dilute HCl, filtered hot, and cooled gave 0.3 g. XVII.HCl, needles, m.  $250-2^{\circ}$  (EtOH). The appropriate bis(halomethyl) compound (1 mole) [or 0.5 mole tetrakis(halomethyl) compound] in CHC13 treated dropwise at  $20-30^{\circ}$  with 2 moles secondary amine in CHCl3, kept at room temperature, and filtered gave the main fraction A; the filtrate evaporated to dryness yielded the main fraction B; both fractions were worked up for product. XIV (5.0 g.), 4.1 g. Bu2NH, and 120 cc. CHC13 kept 2-3 days and evaporated, the residue boiled briefly with 130 cc. EtOH, concentrated to 45 cc., cooled to  $0^{\circ}$ , and the solid filtered off gave 2.8g. (crude) 2,2-dibutyl-1,3-dihydropyrrolo[3,4-b]quinoxalinium bromide (XVIII); the filtrate concentrated to 25 cc. and the precipitate extracted with 40 cc. hot

dioxane left an addnl. 0.9-0.95 g. crude XVIII; the combined crude XVIII recrystd. from EtOH yielded XVIII.0.5H2O, needles, m. 205-6° (decomposition) (EtOH). XIV (3.0 g.), 1.65 g. piperidine, and 60 cc. CHCl3 allowed to stand 2 hrs. and filtered gave 1.8 g. fraction A which recrystd. from absolute EtOH yielded 1,3-dihydropyrrolo[3,4-b]quinoxaline-2-spiropiperidinium bromide (XIX), m. 266-8° (decomposition) (absolute EtOH); the CHCl3 filtrate evaporated, the residual fraction B dissolved in H2O, the solution treated with aqueous NaOH, extracted with CHCl3, the extract dried, diluted with

Et20, filtered, and the filtrate treated with alc. picric acid gave 1.6 g. picrate analog (XX) of XIX, yellow needles, m.  $189-90^{\circ}$  (decomposition) (EtOH). XIV (4.0 g.), 2.2 g. morpholine, and 80 cc. CHCl3 allowed to stand 7 hrs. gave 4.0 g. fraction A consisting of equal parts morpholinium analog (XXI) of XIX and morpholine-HBr; fraction A recrystd. 4 times from 90% EtOH yielded XXI.0.5H2O, needles, m.  $258^{\circ}$ , and morpholine-HBr,

needles, m. 210-13°; fraction A in boiling EtOH treated with hot alc. picric acid gave the picrate analog (XXII) of XXI, yellow needles, m.  $254\text{--}5\,^{\circ}$  (aqueous EtOH). XXII (0.25 g.) in 50 cc. boiling H2O passed through Dowex II and evaporated yielded 0.14 g. chloride analog of XXII, m.  $257-8^{\circ}$  (absolute EtOH). I (4.0 g.), 3.0 g. piperidine, and 60 cc. CHC13 kept 7 days and evaporated to dryness, the residue refluxed with 50 cc. EtOH and C, and the filtrate treated with picric acid gave 2.7 g. 1,3-dihydropyrrolo[3,4-b]thianaphthene-2-spiropiperidinium picrate (XXIII), m.  $215-16^{\circ}$  (EtOH). I (6.0 g.), 3.5 g. morpholine, and 130 cc. CHC13 allowed to stand 1-2 days and filtered gave 2.9 q. fraction A; a 1.6-g. portion in EtOH treated with excess picric acid gave 0.7 g. morpholinium analog of XXIII, m. 226-7° (EtOH). XII (2.3 g.), 3.0 g. piperidine, and 50 cc. CHCl3 kept overnight gave 2.95 g. fraction A; a 1.0-g. portion in MeOH treated with excess alc. picric acid yielded 1.6 g. 2,2,5,5-bis(pentamethylene)-1,3,4,6-tetrahydrobenzo[1,2-c;4,5c']dipyrrolium dipicrate (XXIV), deep yellow needles, m. 282-3° (H2O). XII (5.0 g.), 6.5 g. morpholine, and 100 cc. CHCl3 gave similarly during 24 hrs. 6.8 g. fraction A; a 6.6-g. portion in 400 cc. MeOH with excess picric acid in MeOH gave 6.8 g. bis(3-oxapentamethylene) analog (XXV) of XXIV, needles, m. 290° (decomposition) (H2O). Crude XXV (2.84 g.) in 1000 cc. boiling H2O passed through Dowex II, evaporated in vacuo, and the crude residue (1.37 g.) recrystd. from MeOH-EtOH yielded the hygroscopic chloride dihydrate analog of XXV, did not melt. 40197-27-1P, 1H-Pyrrolo[3,4-b]quinoxalinium, 2,2-dibutyl-2,3-dihydro-, bromide 108875-29-2P, 2H-Pyrrolo[3,4-b]quinoxaline, 2-butyl-1,3-dihydro-108877-45-8P , 2H-Pyrrolo[3,4-b]quinoxaline, 1,3-dihydro-2-propyl-108990-39-2P, 2H-Pyrrolo[3,4-b]quinoxaline, 2-allyl-1,3-dihydro-109567-85-3P, 2H-Pyrrolo[3,4-b]quinoxaline, 2-allyl-1,3-dihydro-, picrate 109655-08-5P, 2H-Pyrrolo[3,4-b]quinoxaline, 2-benzyl-1,3-dihydro-RL: PREP (Preparation) (preparation of) 40197-27-1 CAPLUS 1H-Pyrrolo[3,4-b]quinoxalinium, 2,2-dibutyl-2,3-dihydro-, bromide (1:1)

(CA INDEX NAME)

ΙT

RN

CN

• Br-

RN 108875-29-2 CAPLUS CN 1H-Pyrrolo[3,4-b]quinoxaline, 2-butyl-2,3-dihydro- (CA INDEX NAME)

RN 108877-45-8 CAPLUS

RN 108990-39-2 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxaline, 2,3-dihydro-2-(2-propen-1-yl)- (CA INDEX NAME)

RN 109567-85-3 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxaline, 2,3-dihydro-2-(2-propen-1-yl)-, compd. with 2,4,6-trinitrophenol (1:1) (CA INDEX NAME)

CM 1

CRN 108990-39-2 CMF C13 H13 N3

 $\begin{array}{c|c} & \text{CH}_2\text{-CH} = \text{CH}_2 \\ \hline & \text{N} \end{array}$ 

CM 2

CRN 88-89-1 CMF C6 H3 N3 O7

O<sub>2</sub>N NO<sub>2</sub> OH NO<sub>2</sub>

RN 109655-08-5 CAPLUS

CN 1H-Pyrrolo[3,4-b]quinoxaline, 2,3-dihydro-2-(phenylmethyl)- (CA INDEX NAME)

N CH2-Ph

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ACCESSION NUMBER: 1929:29324 CAPLUS

DOCUMENT NUMBER: 23:29324

ORIGINAL REFERENCE NO.: 23:3472e-i,3473a-c

TITLE: Action of o-phenylenediamines upon dihydroxytartaric

acid

AUTHOR(S): Chattaway, Frederick D.; Humphrey, William G.

SOURCE: J Chem. Soc. (1929) 645-51

DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
CI For diagram(s) see printed CA less

GI For diagram(s), see printed CA Issue.

AB When Na dihydroxytartrate is heated with aqueous o-C6H4(NH2)2, 2 mols of the

diamine react with 1 mol. only of the salt, forming quinoxaline-2,3-dicarboxy-o-phenylenediamide (I); Na dihydroxytartrate is only very sparingly soluble in H2O and any excess above 1 mol. remains in suspension unchanged. When the filtered alk, solution is partly neutralized with HCl, I seps. as a colorless crystalline powder, stable in neutral solution and dissolving readily in cold dilute aqueous alkali, from which it is repptd. on addition of a deficiency of acid. It dissolves in hot dilute HCl (1:50), but on cooling, the o-phenylenediamine salt, (II) of quinxaline-2,3-dicarboxylic acid (III) seps; whereas, if it is dissolved in hot moderately concentrated HCl (1:1), III separated on cooling o-phenylenediamine-HCl remaining in solution The II and III may consequently be obtained directly from the original yellow condensation solution, the former by making the solution weakly acid with HCl, and the latter by saturating

it with gaseous HCl. Attempts to acetylate or benzoylate I by the usual methods also cause decomps., with formation of the di-Ac or the di-Bz derivative of o-C6H4(NH2)2. Heated with Ac2O, III yields the anhydide, while dry NH3 on this anhydride in C6H4 suspensions gives the NH4 salt of 3-carbamylquinoxaline-2-carboxylic acid (IV), from which the acid itself may be obtained on acidification. This amic acid is converted into the corresponding imide (V) on being heated above its m. P., and into the Ac derivative of the imide on boiling with Ac20. On being heated above its m. p., III decomps., evolving CO2 and yielding a small quantity (10%) of quinoxaline; better yields (30%) of this base are obtained by heating the NH4 salt of the acid. In common with other N bases, quinoxaline forms a stable, well-crystallized monotetrachloroiodiede. Similarly, Na chloroquinoxaline-2,3-dicarboxy-p-chloro-o-phenylenediamide, from which the p-chloro-o-phenylenediamine salt of 6-chloroquinoxaline-2,3-dicarboxylic acid, and the free acid (VI) are obtained by heating with dilute and with concentrated HCl, resp. p-Bromo-o-phenylenediamine gives the corresponding Br derivative These halogen-substituted derivs. are considerably less soluble than the unsubstituted compds., and are therefore more readily prepared and purified; otherwise their reactions are analogous. The following compds. were prepared and characterized: I, m. 184° (decomposition). II, lemon-yellow, m. 186° (decomposition). III, prisms containing 2 mols. H2O of crystallization, m.

190° (decomposition after loss of H2O at 110°); Et ester, C14H14O4N2, prisms, m. 83°; NH4 salt, m. 22O-30°; anhydride, pale yellow prisms decomposing and charring 25O-60°. IV, m. 19O-5° (decomposition). V, pale yellow, m. about 260° (decompose); Ac derivative, leaflets, m. about 220° (decomposition). Quinoxaline mono-tetrachloroiodide, C6H4N2. HICl4, m. 125-30° (decomposition). 6-Chlroquinoxaline-2,3-dicarboxy-p-chloro-o-phenylenediamide, C16H8O2N4Cl2, m. 207° (decomposition) (p-chloro-o-phenylenediamine salt, C16H18O4N4Cl3, m. 205° (decomposition));

6-bromoquinoxaline-2,3-dicarboxy-p-bromo-o-phenylenediamide, m. 198° (decomposition) (p-bromo-o-phenyleneamine salt, m. 199° (decomposition)). VI, m. 175° (decomposition) (anhydride, m. 235-40° (decomposition), Et H ester, m. 159°; di-Et ester, m. 60°; NH4 salt, m. 215-25° (decomposition)). 6-Chloroquinoxaline, m. 60°, 6-Bromoquinoxaline-2,3-dicarboxylic acid, m. 172° (decomposition) (anhydride, m. 235-45° (decomposition), Et H ester, m. 161°, di-Et ester, m. 69°, NH4 salt, m. 235-40° (decomposition)). 6-Bromoquinoxaline, m. 56°. Pyrazinetetracarboxylic acid (by oxidation of the anhydride of III), m. 205° (decomposition), di-K di-H salt is crystalline, tetra-Et ester, m.  $104^{\circ}$ . 5660-33-3P, 2,3-Quinoxalinedicarboximide 856101-16-1P , 2,3-Quinoxalinedicarboximide, N-acetyl-RL: PREP (Preparation) (preparation of) 5660-33-3 CAPLUS RN CN 1H-Pyrrolo[3,4-b]quinoxaline-1,3(2H)-dione (CA INDEX NAME)

RN 856101-16-1 CAPLUS CN 1H-Pyrrolo[3,4-b]quinoxaline-1,3(2H)-dione, 2-acetyl- (CA INDEX NAME)

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